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Sampling, Characterization, and Evaluation of Midwest Clays for Iron Ore Pellet Bonding

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UNITED STATES DEPARTMENT OF THE INTERIOR

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

cm	centimeter	m	meter
cm/min	centimeter per minute	m^2	square meter
cm^2/g	square centimeter per gram	m^2/g	square meter per gram
cm^3	cubic centimeter	meq	milliequivalent
cm^3/cm^3	cubic centimeter per cubic centimeter	min	minute
cP	centipoise	mm	millimeter
°C	degree Celsius	µm	micrometer
°C/min	degree Celsius per minute	mV	millivolt
g	gram	pct	percent
g/cm^3	gram per cubic centimeter	pct/min	percent per minute
h	hour	ppm	part per million
kg	kilogram	rpm	revolution per minute
km	kilometer	SLM	standard liter per minute
lb	pound	st	short ton

SAMPLING, CHARACTERIZATION, AND EVALUATION OF MIDWEST CLAYS FOR IRON ORE PELLET BONDING

By Larry A. Haas,[1] Jeffrey A. Aldinger,[2] Rolland L. Blake,[3] and Stephen A. Swan[2]

ABSTRACT

The Bureau of Mines, in cooperation with three Minnesota agencies (Department of Natural Resources, Geological Survey, and the Iron Range Resources and Rehabilitation Board), investigated the potential of using glacial lake clay from Minnesota, as well as other areas, as a binder for iron ore pellets.

The best clay-characterizing procedures for predicting binder effectiveness were the cation exchange capacity (CEC) using the methylene blue method and the plate water absorption test (PWAT).

The best clay binder was found in the Red River Valley. However, compared to a typical western bentonite at the 0.5-pct-addition level, addition of about 2 pct Minnesota clay was required to obtain comparable unfired (green) pellet strengths. Soda ash addition only slightly improved the binding properties of Minnesota clay, but considerable improvement was obtained by the addition of a small quantity of water-absorbing organic compounds. For example, with a mixture of 0.5 pct Minnesota clay or paint rock and 0.1 pct pregelatinized starch, the unfired and fired physical pellet properties were about the same as with 0.5 pct of typical western bentonite. The reduction rate at the 40-pct reduction level and the percentage of plus 6.3-mm particles from the reduction disintegration index test were also about the same.

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INTRODUCTION

Historically, the majority of domestic iron ore has been supplied from the Lake Superior region of northern Minnesota and Michigan. This area currently accounts for 96 pct of total U.S. production, with Minnesota and Michigan responsible for 71 and 25 pct, respectively (1).[4] Sixty million short tons of iron ore pellets were shipped from Minnesota in 1979, but as a result of the severity of the recent economic decline, the production for 1985 was estimated at only 33 million st (2).

The availability of relatively cheap iron ore pellets from foreign sources has had a dramatic effect on the U.S. iron ore mining industry. A pelletizing plant was closed in May 1985, partly because of the inability to compete costwise with the rising imports (3). It is imperative that processing costs be reduced if the domestic iron ore industry is to remain competitive with foreign suppliers.

Approximately one-eighth of the pelletizing cost has been attributed to the cost of the bentonite binder. The cost of binder-grade bentonite on the Mesabi Iron Range has tripled since 1974, and the quality has not been consistent (4). Roughly two-thirds of the total binder cost to the plants is due to transportation from Western States to the Mesabi Iron Range. Obviously, a sizable savings could be realized if a substitute source of suitable native clay binder could be located near the pelletizing plants.

The Bureau of Mines, as part of its mission to investigate alternative technology options for processing domestic iron ores, conducted research to determine if clay from deposits located near the Mesabi Iron Range could be completely or partly substituted for bentonites obtained from the Western States. Not only would such substitution reduce costs, it also would conserve the limited quantities of high-quality bentonite that are economically minable. Some research (5-6) was done in the past using local Minnesota clays as a binder in iron ore pelletization, but to date no binders have been found to replace western bentonites.

Some of the benefits derived from the use of bentonite in the iron ore pelletizing process are as follows.

1. Absorb excess water from the filter cake concentrate to facilitate balling.
2. Provide sufficient drop and compressive strengths to the unfired (green) pellets so that they will withstand screening and transfer operations enroute to the indurating furnace.
3. Moderate the moisture release from the pellets during the drying stage (ambient to 250° C), thereby prevent cracking of pellets due to excessive internal steam pressure.
4. Provide adequate dry pellet strength during the preheat stage, i.e., 250° to 900° C, where pellets are the weakest, until sufficient presintering and/or oxidation bonding occurs.
5. Improve the mechanical properties of the fired (about 1,300° C) pellets, i.e., compressive strength and abrasion resistance to withstand handling and transportation to lower lake ports.
6. Decrease the quantity of fine particles produced during reduction, thus produce a superior blast furnace feed.

In order to be assured that these binder benefits will be obtained, adequate evaluation methods must be available. Unfortunately, meaningful methods for evaluation of the overall quality of binder materials are presently not available (7). Standardized quality acceptance tests and binder specifications for organic and inorganic materials currently are nonexistent. Tests are needed that are relatively fast, simple, and reproducible.

In general, all taconite companies specify a western bentonite (sodium montmorillonite) with high swelling characteristics because it is known to have

[4]Underlined numbers in parentheses refer to items in the list of references preceding the appendixes.

better binding properties than southern bentonite (calcium montmorillonite). Other specifications that have been suggested are Na_2O and CaO contents, slurry viscosities, gel strengths, moisture contents, particle sizes (colloid content), and grit contents. In 1983, ASTM (8) published the plate water absorption test (PWAT), which is presently the most widely utilized specification for bentonite acceptance.

The goals of this research were (1) to determine the relative usefulness of some clay characterization tests for evaluating different clay binders, and (2) to determine under what conditions local midwestern clays could be used as a binder for iron ore concentrates.

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RAW MATERIALS

MIDWEST CLAY SAMPLES

Four types of natural clay exist in Minnesota: (1) marine kaolinitic clay in the Ordovician Decorah shale, (2) kaolinitic clay from residual weathering during the Cretaceous period of Precambrian igneous and metamorphic rocks, (3) Pleistocene clay in glacial deposits, including clay interstratified in tills and reworked tills and glacial lake clay, that was deposited in six major lakes formed in front of melting continental glaciers when the ice blocked the natural northward drainage of the water, and (4) Holocene clay at the bottom of present-day lakes.

The clays with the most potential as binders would be those easily accessible and those with the highest smectite (montmorillonite) content. The marine clays and the Cretaceous weathering clays are kaolinitic (9). The Holocene clays at the bottom of present-day lakes are scattered, thin, and covered by water or soil. The marine clays are interstratified with shale, and the Cretaceous clays are covered by about 30 m of glacial deposits. Clays in glacial till are intimately mixed with silt, sand, and gravel. The glacial lakes are gone, but their deposits are widespread (fig. 1). Glacial lake clays are exposed at the present surface, and scattered well logs show them to extend in depth to over 30 m. Therefore, glacial lake clays were selected for the major testing studies as a pellet binder. Previously, a few of these samples have shown some smectite content and binder potential (5-6).

Sampling of glacial lake clays in this study involved three methods: (1) Auger samples were obtained by commercial drillers; (2) smaller split-tube samples (2.5- and 7.5-cm diam) were obtained from the Minnesota Department of Transportation, the Minnesota Geological Survey, the North Dakota Geological Survey, and

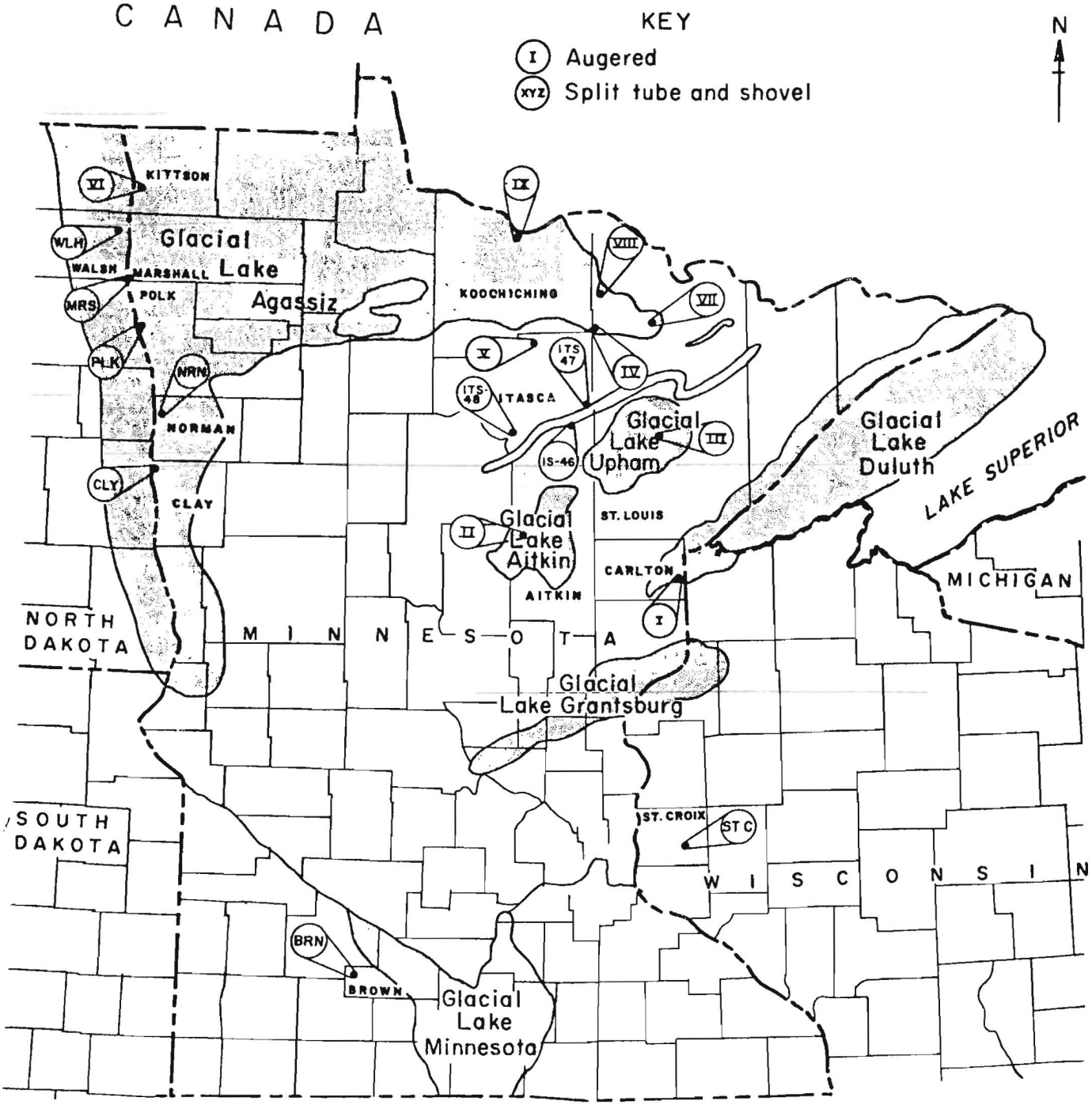

FIGURE 1.—Map of former major glacial lakes of Minnesota and locations of auger, split-tube, and shovel sampling areas.

the U.S. Army Corps of Engineers; and (3) shovel samples were obtained by Bureau personnel from glacial lake clay outcrops and pits. The source locations of the samples are shown in figure 1 and listed in table 1. The area column in table 1 contains Roman numerals for augered samples and county abbreviations for split-tube and shovel samples.

Auger Sampling

Auger sampling areas of glacial lake clays were first chosen within 200 km of the operating taconite plants (areas I through V in figure 1). As the study progressed and the results of clay characterization and pellet strength tests indicated a rather marginal response, other areas were selected (areas VI through IX in figure 1). Specific field augering sites were chosen in each area by a study of maps, aerial photographs, and field notes and experience of the geologists. Sites were chosen along Federal, State, county, and township roads crossing public lands for easy access; permission was obtained in each case. Well logs were useful in avoiding known aquifers, and none were penetrated. Auger holes were abandoned according to regulations of the Minnesota Department of Health.

The 6-in-diam augering operation consisted of removing 5 ft section samples sequentially to depths up to 75 ft. Each 5-ft section (about 30 kg) was kept separate and placed first in a plastic bag and then a cloth bag; a 0.1-kg sample was also placed in a separate plastic bag for an as-received moisture determination. All of the 5-ft samples were spread out on laboratory benches, and several samples from each hole were selected for the binder characterization and pelletizing tests. These grayish or reddish selected samples (table 2) consisted of three or four 5-ft section samples from each auger hole, which were essentially free of grit, pebbles, peat, and soil; and that were located nearest the top, middle (one or two samples), and bottom of the hole.

The material in each 5-ft selected section was subdivided into about 1-in-diam pieces and placed randomly in a pile. About one-fifth slice of the pile was dried at 75° C overnight, and the remainder was placed in a sealed bag. The dried pieces were ground to minus 1/4-in in a gyratory cone crusher and pulverized in a 8-in-diam Raymond mill.[5] The chemical analyses of the augered samples are shown in table 3.

Split-Tube and Shovel Sampling

Several split-tube and shovel samples (about 1 kg each) were obtained during this study. The location of the sampling sites was based on information from the U.S. Department of Agriculture Soil Conservation Service, Minnesota Department of Transportation, and other sources. These subsoil samples were dried and pulverized by the same methods as the augered samples. The chemical analyses of these samples are shown in table 4.

COMMERCIAL SAMPLES

Various materials were obtained from commercial sources for the clay characterization and the pellet binder studies. These powdered materials were dried by the same methods as the other samples. Chemical analyses of the commercial samples are given in table 5.

The commercial organic materials were used as received. The carboxyl methyl cellulose (CMC) samples, designations of P and H, had sodium contents of 20 and 9 pct, respectively. Polyacrylate (PAC) and starch-graft copolymer (SGC), type J, also contained 20 pct sodium. The sodium content of the starch-graft copolymer, type A, was 11 pct; the other organic materials contained less than 3 pct sodium. The potassium and sulfur contents of all the organic samples were less than 0.3 and 0.05 pct, respectively. The pregelatinized starch (PS), No. 300, had the highest phosphorus content of 2.7 pct. The guar gums (GG) phosphorus contents ranged from 0.06 to 0.2 pct, but all the other organics contained less than 0.06 pct phosphorus.

[5]Reference to specific products does not imply endorsement by the Bureau of Mines.

TABLE 1. - Location of samples obtained for clay characterization studies

County, by State	Township number	Range	Section		Area[1]	Hole	Sampling method
			Number	Quarter			
Minnesota:							
Aitkin (AKN).....	47	26	2	SW	II	35	Auger.
	47	26	10	NE	II	34	Do.
	48	26	4	SW	II	28	Do.
	48	26	4	SE	II	29	Do.
	48	26	15	SW	II	30	Do.
	48	26	35	SE	II	32	Do.
	48	26	36	NW	II	33	Do.
Brown (BRN).....	109	34	NA	NA	BRN	43	Shovel.
Carlton (CRT).....	46	17	16	NW	I	1	Auger.
	46	17	15	NE	I	2	Do.
	46	17	15	NE	I	3	Do.
	46	17	15	NE	I	4	Do.
	46	17	13	NW	I	5	Do.
	47	16	16	SE	I	7	Do.
	46	16	6	SE	CRT	44	Split tube.
Clay (CLY).....	142	48	32	NW	CLY	45	Do.
Itasca (ITS).....	56	23	1	SE-NE	ITS	46	Shovel.
	57	22	24	SE	ITS	47	Split tube.
	62	26	21	SE	V	18	Auger.
	62	26	28	NW	V	19	Do.
	62	26	20	SW	V	20	Do.
	62	26	30	NE	V	21	Do.
	62	26	24	SE	V	22	Do.
	62	26	24	SW	V	23	Do.
	62	26	21	NW	V	25	Do.
	55	26	NA	NA	ITS	48	Shovel.
Kittson (KIT).....	161	50	9	NW	VI	36	Auger.
	161	50	9	NW	VI	37	Do.
Koochiching (KCH).....	70	26	32	SE	IX	40	Do.
	70	25	32	SE	IX	41	Do.
Marshall (MRS).....	154	50	6	NW	MRS	49	Split tube.
Norman (NRM).....	144	48	6	NW	NRM	50	Do.
Polk (PLK).....	151	50	2	NA	PLK	51	Shovel.
St. Louis (STL).....	54	18	9	NW	III	9	Auger.
	54	18	10	SW	III	8	Do.
	54	18	21	SW	III	10	Do.
	54	18	22	NW	III	11	Do.
	55	18	21	SE	III	12	Do.
	62	21	27	SW	IV	13	Do.
	62	21	22	SW	IV	14	Do.
	62	21	16	NE	IV	15	Do.
	62	21	19	NE	IV	16	Do.
	62	21	19	NE	IV	17	Do.
	62	18	5	NW	VII	38	Do.
	63	21	9	NE	III	39	Do.
North Dakota: Walsh (WLH).	158	51	34	NE	WLH	52	Split tube.
Wisconsin: St. Croix (STC)	29	15	32	NW-SW	STC	53	Shovel.

NA Not available.

[1]The Roman numerals refer to different augering sites and the letter abbreviations to different counties.

TABLE 2. - Evaluation of as-received Minnesota selected augered samples

Sample	Auger section, ft	H_2O, pct[1]	Pebbles	Color[2]	Texture[3]	Strength,[4] lb	Shrink,[5] pct
Area I:							
1A	10-15	24.8	None	Gray	Sm	122	16.5
1B	25-30	39.2	..do	..do	Sl	119	29.8
1C	45-50	26.2	Some	..do	Sl	132	11.6
1D	70-75	26.1	None	..do	Sl	129	16.5
2A	5-10	25.4	..do	Red	Sl	144	17.6
2B	25-30	24.6	..do	Gray	Sl	142	19.0
2C	70-75	28.4	..do	..do	Sl	105	14.3
3A	15-20	24.1	..do	..do	Sm	135	18.6
3B	45-50	26.9	Some	..do	Sl	101	14.6
3C	70-75	26.3	None	..do	Sl	207	19.0
4A	0- 5	26.6	..do	Red	R	172	16.5
4B	20-25	33.1	..do	Red	Sm	76	20.4
4C	35-40	24.9	..do	Gray	Sm	144	13.1
4D	70-75	26.7	..do	..do	Sm	160	14.3
5A	5-10	20.6	..do	..do	Sm	151	22.3
5B	30-35	28.5	..do	..do	Sm	112	20.1
5C	50-55	16.9	..do	..do	Sm	154	7.1
7A	15-20	35.2	..do	..do	Sm	125	24.3
7B	40-45	18.3	..do	..do	Sm	69	5.9
7C	60-65	38.7	..do	Red	Sm	101	29.4
7D	72-75	33.0	..do	Gray	Sm	98	27.3
Area II:							
32A	5-10	24.2	..do	..do	G	102	12.0
32B	30-35	3.6	..do	..do	Sm, R	38	7.1
32C	45-50	31.0	..do	..do	Sm, R	149	16.1
33A	15-20	19.5	..do	..do	Sm	127	11.3
33C	45-50	24.8	..do	..do	Sm	140	13.9
Area III:							
8A	15-20	15.0	Many	..do	Sm, R	102	7.8
9B	25-30	24.6	None	..do	Sm	96	10.9
10A	10-15	19.8	Some	..do	G, Sm	77	9.0
10B	15-20	13.6	..do	..do	Sm	121	4.4
11A	10-15	26.2	None	..do	Sm	75	16.1
12A	15-20	17.7	..do	..do	Sm	92	13.5
Area IV:							
13A	15-20	24.7	..do	..do	Sm	178	20.4
13B	30-35	31.6	..do	..do	Sm	96	24.0
14A	5-10	23.0	..do	..do	Sm	60	18.3
14B	30-35	30.7	..do	..do	Sm	128	15.0
15A	5-10	25.4	..do	..do	Sm	100	13.1
15B	20-25	27.7	..do	..do	Sm	135	17.2
15C	45-50	30.6	..do	..do	Sm	108	16.1
16A	5-10	25.7	Some	..do	R	97	12.4
16B	20-25	33.4	None	..do	Sm	121	19.7
16C	40-45	30.2	..do	..do	Sm	97	17.2
17A	5-10	25.7	Many	..do	Sm	164	13.9
17B	20-25	NE	..do	..do	G	71	7.1

See explanatory notes at end of table.

TABLE 2. - Evaluation of as-received Minnesota selected augered samples--Continued

Sample	Auger section, ft	H_2O, pct[1]	Pebbles	Color[2]	Texture[3]	Strength,[4] lb	Shrink,[5] pct
Area V:							
18A	10-15	19.2	Some	Gray	Sm	177	8.2
18C	30-35	19.4	..do	Red	Sm	152	19.4
19A	5-10	18.4	..do	Gray	Sm	166	10.5
19B	20-25	15.4	..do	..do	Sm	98	8.6
19C	30-35	19.1	..do	..do	Sm	156	10.1
20A	5-10	16.8	..do	Red	Sm	174	8.6
20B	15-20	18.5	..do	Red	Sm	134	11.6
20C	25-30	17.5	..do	Red	Sm	126	9.9
21A	5-10	15.2	..do	Gray	Sm, R	180	7.5
21B	45-50	21.0	..do	..do	Sm, R	207	11.3
22A	5-10	16.7	..do	Red	G, Sm	110	13.5
22B	15-20	16.5	..do	Gray	Sm	137	13.1
22C	40-45	17.1	..do	..do	Sm	173	13.5
23A	10-15	16.6	..do	..do	Sm	144	8.2
23B	25-30	16.2	..do	..do	Sm	157	11.3
23C	40-45	17.3	..do	..do	Sm	160	9.4
25A	25-30	NE	..do	..do	Sm	-	12.8
Area VI:							
36A	15-20	31.0	None	Red	Sm	174	17.9
36B	30-35	32.9	..do	Gray	Sm	111	27.1
36C	70-75	31.3	..do	..do	Sm	167	26.7
37A	25-30	33.3	..do	..do	Sm	147	22.2
37B	45-50	34.0	..do	..do	Sm	97	26.4
37C	70-75	31.3	..do	..do	Sm	125	21.5
Area VII:							
38A	5-10	26.2	..do	..do	Sm	127	15.7
38B	25-30	30.2	..do	..do	Sm	49	16.1
38C	45-50	33.1	..do	..do	Sm	92	22.9
Area VIII:							
39A	15-20	28.4	Some	..do	Sm	49	11.6
39B	30-35	21.7	..do	..do	Sm, R	251	10.9
39C	45-50	27.9	..do	..do	Sm	137	19.0
Area IX:							
40A	15-20	17.3	..do	..do	Sm, G	85	13.5
40B	45-50	18.3	..do	..do	Sm	171	12.8
41A	15-20	24.6	..do	..do	Sm	52	3.2
41B	45-50	20.0	..do	..do	Sm	163	14.6

NE Not evaluated.

[1]Percentage weight loss at 105° C after 24 h.

[2]Munsel colors 5 yr 5/1 (gray) and 10 R 3/6 (dusty red).

[3]G, gritty; R, resistant; Sm, smooth; and Sl, slippery.

[4]Unconfined dry compressive strength of cylindrical briquets (1/2-in diam and 5/8-in height.

[5]Briquet diameter shrinkage with an initial water content of about 20 pct and dried at 105° C for 24 h.

TABLE 3. - Chemical analyses of augered clay samples[1]

Sample	Auger section, ft	Si-Al ratio	Partial analyses, pct											LOI at indicated temp, pct			
			Si	Al	Ca	Mg	Fe	Ti	K	Na	C	P	S	105° C	260° C	510° C	1,000° C
Area I:																	
1A.....	10-15	2.44	21.5	8.8	6.8	2.8	3.0	0.41	1.4	0.6	3.2	0.12	0.04	0.61	1.80	4.70	14.50
1B.....	25-30	2.40	22.1	9.2	3.2	2.4	4.4	.49	2.0	.7	1.1	.10	.04	2.00	2.70	5.20	9.40
1C.....	45-50	3.01	23.8	7.9	5.8	2.5	2.8	.42	1.6	.8	2.5	.11	.15	1.20	1.60	3.50	11.70
1D.....	70-75	3.01	25.0	8.3	4.4	2.3	3.1	.46	1.7	1.0	2.3	.10	.14	.92	1.70	3.70	9.90
2A.....	5-10	2.21	21.2	9.6	3.8	2.5	4.4	.48	2.2	.8	1.2	.10	.02	2.10	3.10	5.60	11.00
2B.....	25-30	2.88	24.8	8.6	4.4	2.6	3.5	.46	1.8	1.0	1.9	.10	.08	1.50	2.00	4.10	10.50
2C.....	70-75	3.02	25.4	8.4	4.3	2.3	3.3	.46	1.7	1.0	1.9	.10	.11	1.30	1.90	4.20	10.30
3A.....	15-20	3.07	25.5	8.3	4.3	2.2	3.3	.47	1.8	1.0	1.9	.11	.12	1.20	1.90	3.90	10.00
3B.....	45-50	2.96	24.6	8.3	5.1	2.5	2.9	.41	1.8	.6	2.0	.10	.03	1.40	2.10	4.00	11.10
3C.....	70-75	2.93	24.0	8.2	5.9	2.8	2.8	.42	1.6	.9	3.5	.10	.19	1.10	1.60	2.80	12.10
4A.....	0- 5	2.47	23.2	9.4	4.2	2.7	4.1	.47	2.1	.7	1.5	.08	.01	2.10	3.40	5.90	11.80
4B.....	20-25	2.36	22.9	9.7	3.1	2.8	4.8	.50	2.2	.7	1.0	.09	.02	2.30	3.40	6.10	10.30
4C.....	35-40	3.05	24.7	8.1	6.1	2.9	2.7	.42	1.6	1.0	3.2	.10	.20	1.20	1.70	3.70	12.80
4D.....	70-75	3.11	25.8	8.3	4.1	2.3	3.3	.48	1.7	1.0	1.9	.10	.11	1.20	2.00	3.90	10.40
5A.....	5-10	2.62	22.8	8.7	5.1	2.8	3.5	.45	2.6	.8	1.7	.11	.31	2.00	3.90	10.40	12.90
5B.....	30-35	2.47	23.0	9.3	3.4	2.9	4.1	.49	2.6	.8	1.5	.10	.06	1.20	2.10	5.20	10.10
5C.....	50-55	3.23	26.2	8.1	3.7	2.2	3.2	.49	2.5	.8	1.8	.10	.10	.62	1.50	3.50	8.10
7A.....	15-20	2.15	21.3	9.9	4.0	2.8	4.6	.49	3.1	.7	1.4	.10	.01	1.80	3.00	6.40	11.50
7B.....	40-45	3.29	23.7	7.2	8.4	3.2	1.8	.34	1.3	.8	5.0	.13	.30	.26	1.10	1.70	16.70
7C.....	60-65	2.51	22.6	9.0	3.6	2.8	4.5	.50	3.0	.7	1.4	.11	.04	1.30	2.40	5.30	9.70
7D.....	72-75	2.69	23.4	8.7	3.8	2.7	4.2	.52	2.5	.9	1.9	.11	.08	1.20	2.00	4.60	9.70
Area II:																	
32A.....	5-10	3.23	25.5	7.9	6.8	2.7	2.7	.39	1.8	1.1	1.7	.12	.03	2.00	2.10	4.20	9.20
32C.....	45-50	2.67	24.0	9.0	3.6	2.1	3.0	.42	1.6	.7	3.4	.09	.16	1.20	2.20	4.30	14.60
33A.....	15-20	2.78	23.6	8.5	6.9	2.8	2.7	.39	1.6	1.2	2.5	.12	.05	.75	1.50	3.70	10.20
33B.....	30-35	3.12	25.9	8.3	4.7	2.2	2.7	.41	1.4	1.0	3.5	.10	.22	.38	1.20	3.70	12.50
33C.....	45-50	2.94	23.5	8.0	6.7	2.7	2.3	.38	1.5	.8	3.7	.12	.16	1.20	1.60	3.50	14.80
Area III:																	
8A.....	15-20	3.88	28.3	7.3	3.9	1.9	2.3	.35	1.4	1.4	1.4	.09	.11	.69	.96	2.10	6.70
9A.....	8-10	3.88	28.3	7.3	4.8	2.2	2.2	.38	1.4	1.7	2.3	.13	.20	.79	1.10	1.90	7.70
9B.....	25-30	2.91	25.6	8.8	3.0	2.1	3.4	.43	1.8	1.5	1.2	.09	.04	1.30	1.80	3.50	7.50
10A.....	10-15	3.85	28.9	7.5	3.6	2.0	2.4	.37	1.4	1.4	1.5	.11	.08	1.10	1.50	2.80	7.10
10B.....	15-20	3.99	29.1	7.3	3.4	1.8	2.4	.37	1.4	1.5	1.5	.09	.10	.85	1.20	2.80	5.70
11A.....	10-15	3.06	26.0	8.5	3.1	2.0	3.3	.43	1.6	1.4	1.2	.11	.02	1.70	2.30	3.80	7.50
12A.....	15-20	3.41	27.6	8.1	2.4	1.9	3.8	.47	1.7	1.8	.7	.10	.05	.83	4.50	6.80	7.50
Area IV:																	
13A.....	15-20	2.65	23.6	8.9	5.6	2.7	3.0	.41	1.7	1.1	2.8	.11	.05	.50	1.60	5.90	12.80
13B.....	30-45	2.39	22.7	9.5	6.1	2.8	3.2	.41	1.8	.7	2.9	.11	.05	2.70	5.60	6.90	15.10
14A.....	5-10	2.76	24.0	8.7	5.6	2.5	2.9	.40	1.7	1.2	2.5	.12	.01	1.90	2.30	4.50	11.90
14B.....	30-35	2.40	22.1	9.2	8.8	2.8	3.0	.40	1.8	.8	3.3	.11	.05	2.30	3.00	3.20	14.90
15A.....	5-10	2.43	22.1	9.1	7.3	2.7	2.8	.40	2.2	1.2	2.4	.12	.01	NE	2.50	5.70	14.30
15B.....	20-25	2.28	21.4	9.4	7.0	2.7	3.0	.39	2.1	1.0	2.4	.10	.02	NE	2.90	6.60	14.90
15C.....	45-50	2.43	21.9	9.0	6.9	2.5	3.0	.39	2.1	1.2	2.7	.10	.02	NE	2.40	5.70	13.90

See Explanatory notes at end of table.

TABLE 3. - Chemical analyses of augered clay samples[1]--Continued

Sample	Auger section, ft	Si-Al ratio	Partial analyses, pct											LOI at indicated temp, pct			
			Si	Al	Ca	Mg	Fe	Ti	K	Na	C	P	S	105° C	260° C	510° C	1,000° C
Area IV--Con.																	
16A.....	5-10	2.48	21.6	8.7	7.7	2.8	2.6	0.38	2.1	1.3	2.9	0.10	0.01	NE	2.20	5.00	14.50
16B.....	20-25	2.38	22.4	9.4	5.8	2.3	3.2	.41	2.3	1.2	2.3	.11	.05	NE	2.90	6.30	13.20
16C.....	40-45	2.37	22.5	9.5	4.0	2.2	3.9	.44	2.8	1.8	1.3	.08	.03	NE	2.10	5.00	9.30
17A.....	5-10	2.71	24.1	8.9	4.6	1.9	3.1	.40	2.2	1.7	1.4	.10	.00	NE	2.20	4.40	9.50
17B.....	20-25	3.57	28.9	8.1	2.6	1.7	2.4	.36	3.4	2.7	.2	.14	.01	NE	.50	1.20	2.10
Area V:																	
18A.....	10-15	2.67	22.2	8.3	8.3	2.9	2.1	.34	1.6	1.0	2.9	.13	.01	NE	1.70	4.20	15.70
18B.....	20-25	2.57	21.6	8.4	8.4	3.0	2.2	.34	1.7	.9	4.1	.11	.08	NE	1.70	4.50	16.50
18C.....	30-35	2.70	22.1	8.2	8.4	2.9	2.0	.33	1.6	1.0	3.8	.13	.26	NE	1.30	4.00	15.60
19A.....	5-10	2.56	21.8	8.5	8.4	2.9	2.1	.34	1.6	1.0	3.7	.11	.02	NE	1.70	4.40	15.90
19B.....	20-25	3.29	25.0	7.6	8.0	2.8	1.5	.30	1.4	1.4	3.2	.12	.13	NE	.88	2.70	12.30
19C.....	30-35	2.77	22.7	8.2	8.3	2.9	1.9	.33	1.5	1.1	3.5	.11	.16	NE	1.20	3.80	14.70
20A.....	5-10	2.73	22.9	8.4	8.4	3.0	2.0	.34	1.6	1.0	4.4	.13	.05	NE	1.50	4.40	15.90
20B.....	15-20	2.74	22.5	8.2	8.3	3.1	1.9	.34	1.2	.8	4.1	.13	.13	1.10	1.60	3.80	16.10
20C.....	25-30	2.82	23.1	8.2	7.9	3.0	1.9	.33	1.3	.9	4.3	.12	.15	.99	1.50	5.60	16.20
21A.....	5-10	2.86	22.9	8.0	8.1	3.1	2.0	.33	1.2	.8	4.1	.14	.01	1.50	2.10	4.00	23.10
21B.....	45-50	2.60	21.8	8.4	8.1	3.2	2.1	.34	1.3	.7	4.2	.12	.15	1.50	2.10	4.70	16.90
22A.....	5-10	3.06	23.9	7.8	7.9	3.1	1.9	.32	1.2	.9	3.9	.13	.01	1.30	1.80	3.60	14.60
22B.....	15-20	2.90	23.5	8.1	8.1	3.2	1.9	.33	1.2	.8	4.2	.12	.14	1.20	1.70	3.60	15.60
23A.....	10-15	3.06	24.2	7.9	7.9	3.0	1.8	.33	1.3	.8	3.9	.13	.04	.93	1.20	3.20	14.40
23B.....	25-30	2.96	23.4	7.9	8.0	3.0	1.9	.33	1.2	.8	4.4	.12	.14	.78	1.30	3.10	14.80
23C.....	40-45	2.85	23.1	8.1	8.0	3.0	1.9	.33	1.2	.8	5.0	.13	.09	.58	1.00	3.00	15.10
25A.....	25-30	2.85	23.1	8.1	8.0	3.0	1.9	.33	1.3	.8	4.9	.11	.13	.82	1.20	3.20	15.90
Area VI:																	
36A.....	15-20	2.57	25.2	9.8	2.5	1.9	4.0	.48	1.9	.5	.9	.09	.22	4.00	5.70	7.90	12.90
36B.....	30-35	2.35	24.0	10.2	2.6	1.9	4.2	.47	1.9	.4	1.8	.08	.11	3.90	5.40	8.60	13.80
36C.....	70-75	2.73	25.1	9.2	3.5	2.0	3.6	.44	2.0	.5	1.5	.10	.13	4.20	6.60	9.10	15.90
37A.....	25-30	2.54	24.4	9.6	2.9	1.9	3.8	.47	2.0	.5	1.5	.10	.11	3.40	4.90	7.80	13.70
37B.....	45-50	2.51	24.3	9.7	3.1	1.9	3.9	.46	1.9	.4	1.7	.09	.11	3.50	5.00	8.10	14.00
37C.....	70-75	2.82	25.4	9.0	3.8	2.1	3.5	.44	1.9	.5	1.9	.10	.17	3.00	4.30	6.90	14.00
Area VII:																	
38A.....	5-10	3.03	21.2	7.0	5.4	2.4	3.2	.41	2.0	1.2	2.4	.10	.02	2.80	3.70	7.80	13.90
38B.....	25-30	3.06	21.1	6.9	6.5	2.7	3.1	.40	2.0	1.2	2.8	.12	.04	2.30	3.30	6.40	14.40
38C.....	45-50	2.94	20.3	6.9	7.1	2.8	3.1	.39	1.8	.9	1.3	.10	.02	2.70	3.90	7.90	16.50
Area VIII:																	
39A.....	15-20	3.33	21.3	6.4	7.7	2.8	2.6	.37	1.8	1.3	3.1	.11	.08	3.20	4.20	7.10	16.80
39B.....	30-35	3.35	22.1	6.6	6.7	2.7	2.6	.37	1.8	1.4	2.6	.11	.07	1.40	2.20	4.70	12.60
39C.....	45-50	3.10	20.8	6.7	7.1	2.7	2.9	.38	1.8	1.1	3.1	.11	.12	2.40	3.20	6.30	15.40
Area IX:																	
40A.....	15-20	3.12	18.1	5.8	13.0	3.2	1.9	.32	1.5	.8	4.1	.15	.09	1.50	2.00	3.90	18.00
40B.....	45-50	3.38	20.3	6.0	9.3	3.0	1.9	.32	1.5	.8	3.6	.14	.13	1.70	2.00	4.30	16.10
41A.....	15-20	4.09	22.1	5.4	8.5	3.2	1.7	.32	1.4	1.0	4.0	.14	.06	1.30	1.70	3.00	16.90
41B.....	45-50	3.41	20.1	5.9	9.2	3.0	1.9	.32	1.5	.9	3.8	.13	.12	1.50	2.10	4.30	15.80

LOI Loss on ignition between indicated temperature and 75° C.
NE Not evaluated.
[1]Mn percentage <0.1 pct.

TABLE 4. - Chemical analyses of split-tube and shovel clay samples

Sample[1]	Si-Al ratio	Partial analyses, pct											LOI at indicated temp, pct			
		Si	Al	Ca	Mg	Fe	Ti	K	Na	C	P	S	105° C	260° C	510° C	1,000° C
BRN-43...	3.88	32.2	8.3	0.3	0.6	2.7	NE	1.6	0.3	4.6	NE	0.09	NE	NE	NE	7.3
CRT-44...	2.87	23.8	8.3	7.2	2.7	7.7	0.4	2.0	1.0	4.1	0.12	.19	NE	1.8	3.4	14.8
CLY-45...	3.21	24.7	7.7	3.9	2.0	3.5	.4	1.7	.6	2.7	.10	.08	2.3	4.0	7.8	13.5
ITS-46...	1.26	3.4	2.7	.2	1.6	48.7	.5	<.1	.2	.6	.15	.01	2.5	6.9	10.7	12.1
ITS-47...	2.62	25.4	9.7	.3	1.3	4.3	.5	1.3	<.1	1.8	.07	.35	1.2	3.9	8.0	10.1
ITS-48...	3.16	24.3	7.7	1.1	1.8	4.4	.5	2.0	1.1	1.0	.07	.02	3.9	5.9	8.2	9.9
MRS-49...	3.77	24.5	6.5	6.0	2.4	2.5	.4	1.5	.6	4.0	.10	.34	1.6	2.3	6.4	14.8
NRM-50...	2.90	22.9	7.9	3.3	1.9	4.2	.4	1.8	.4	2.9	.09	.13	1.1	4.2	8.4	13.3
PLK-51...	2.72	23.4	8.6	7.3	3.0	2.6	.4	1.4	.4	3.3	.12	.26	1.7	2.5	7.0	17.0
WLH-52...	3.38	24.0	7.1	3.6	1.9	3.5	.4	1.6	.5	3.0	.10	.19	2.1	2.1	6.1	11.3
STC-53...	3.83	30.2	7.9	1.3	1.3	7.9	.5	1.0	.4	.2	.05	<.01	NE	1.9	3.2	5.6

LOI Loss on ignition between indicated temperature and 75° C.

NE Not evaluated.

[1]See table 1.

TABLE 5. - Chemical analyses of commercial inorganic samples

Sample	Si-Al ratio	Partial analyses, pct											LOI at indicated temp, pct			
		Si	Al	Ca	Mg	Fe	Ti	K	Na	C	P	S	105° C	260° C	510° C	1,000° C
Chlorite, CH.....	0.78	9.4	12.0	<0.1	4.3	17.6	0.5	<0.1	<0.1	0.58	<0.01	<0.01	<0.1	0.2	1.4	8.5
Meta bentonite, M-B.....	1.57	20.9	13.3	5.4	2.5	1.4	.6	4.8	.3	1.80	.08	.01	1.4	2.1	4.8	12.4
Silica gel, SG.....	>9.99	40.5	<.2	<.1	<.1	<.1	<.1	<.1	<.1	.56	<.01	.05	5.9	6.4	9.3	11.3
Zeolite, Z.....	1.05	17.2	16.4	<.1	<.1	.2	<.1	.1	11.6	<.10	<.01	.02	4.4	13.8	15.4	17.5
Kaolinite, KA-1.....	.98	21.3	21.7	<.1	<.1	.4	.4	.1	.1	.27	<.01	.04	.2	.4	3.0	4.4
Vermiculite, VE.....	2.33	9.8	4.2	10.2	9.0	.4	<.1	<.1	<.1	8.60	.07	.02	1.9	2.6	14.2	34.3
Fire clay, KA-2.....	2.09	27.4	13.1	.1	.8	1.2	.8	1.7	.1	1.60	.03	.14	1.1	1.8	7.9	9.7
Attapulguite, AT.....	3.74	25.4	6.8	2.1	3.2	2.4	.4	.4	<.1	1.30	.16	.12	6.2	9.1	14.1	16.1
Illite, I-S.....	2.51	25.9	10.3	.4	1.6	4.5	.6	3.8	.4	2.00	.13	.25	1.5	2.6	7.1	8.9
Al_2O_3-colloidal, Al-C....	<.01	.1	35.4	1.4	<.1	<.1	0.1	0.1	.1	4.50	0.01	.05	5.5	12.1	27.4	29.6
Beidellite, BE.....	2.55	25.8	10.1	.7	1.5	4.3	.5	1.7	.8	.44	0.01	.02	5.0	6.1	10.0	10.9
Southern bentonite, SB...	3.53	28.6	8.1	2.0	1.7	2.3	.3	.6	.2	1.40	.03	.22	6.8	8.5	11.5	14.5
Bentonite-mica, B-M.....	2.54	22.9	9.0	.3	1.3	3.1	0.2	.1	1.9	1.50	0.01	.13	8.1	8.7	9.6	14.2
Western Bentonite:																
WB-3.....	4.85	32.5	6.7	1.0	1.6	1.0	.2	.2	1.5	.13	.05	.11	6.2	6.3	7.9	11.0
WB-4.....	4.68	33.2	7.1	1.0	1.0	2.2	.2	.4	1.5	.45	0.01	.04	2.6	3.7	4.3	7.9
WB-5.....	4.29	32.2	7.5	1.4	1.2	2.4	.2	4.0	1.3	.25	0.01	.06	2.8	4.6	5.3	9.8
WB-6.....	2.72	28.8	10.6	1.4	1.4	3.2	0.3	.4	1.4	.83	0.01	.15	1.4	NE	NE	7.1
WB-7.....	3.62	29.7	8.2	1.3	1.1	2.5	.3	.4	2.2	1.10	0.01	.05	4.6	5.4	6.3	9.8
WB-8.....	2.41	25.3	10.5	2.7	1.7	2.9	.2	.6	1.7	1.80	.03	.14	4.4	5.9	7.4	14.4
WB-9.....	3.01	25.6	8.5	2.2	0.9	4.4	.3	.3	2.0	1.70	.17	.08	6.7	8.6	7.4	13.1
WB-10.....	3.38	29.4	8.7	1.2	1.0	3.1	NE	.4	1.9	.38	NE	.15	5.0	5.7	6.6	10.5
WB-11.....	3.87	30.6	7.9	1.0	1.2	3.5	.2	.3	.9	.29	0.01	.03	4.3	6.0	6.6	9.9
WB-12.....	3.13	29.4	9.4	1.1	1.3	2.6	NE	.5	2.1	.29	NE	.15	4.1	4.8	5.6	9.5
WB-13.....	2.72	28.0	10.3	.9	1.4	2.8	NE	.4	1.7	.27	NE	.18	5.5	6.2	7.0	11.5
WB-14.....	3.01	28.3	9.4	.9	1.3	2.6	.2	.4	1.6	.28	0.01	.18	2.9	4.5	5.1	9.7
WB-15.....	3.18	28.3	8.9	.7	1.1	3.5	.2	.2	1.5	.80	0.01	.06	5.1	5.5	6.8	9.4
WB-16.....	2.38	25.0	10.5	1.0	1.6	2.5	.2	.2	1.3	.94	0.01	.21	6.5	7.1	8.4	12.5
WB-17.....	2.91	27.9	9.6	1.1	1.5	2.9	.2	.4	1.5	.23	.02	.10	2.2	4.6	5.5	9.7

LOI Loss on ignition between indicated temperature and 75° C.
NE Not evaluated.

IRON ORE CONCENTRATE

The iron ore used in this research was a magnetic taconite concentrate obtained from the eastern Mesabi Range. It was dried at 105° C overnight and screened to minus 28 mesh to remove any foreign material. The chemical analyses of the dried concentrate were Fe_T, 65.2 pct, Fe^{2+}, 21.7 pct, and Si, 2.6 pct. The Blaine surface area and density were 0.17 m^2/g and 4.9 g/cm^3, respectively.

EXPERIMENTAL METHODS

The experimental methods consisted of (1) characterizing the clays by several diagnostic tests and (2) evaluating the clays as binders for agglomerating iron ore concentrate. The clay characterization tests were similar to those used in the iron ore, foundry, and drilling mud industries (10); a description of the less common characterization procedures is given in appendix A. Appendixes B and C describe the unfired and fired pellet preparation and testing methods, which were similar to those used by the iron ore industry, except a dry iron ore concentrate was used in this research as the starting material.

EXPERIMENTAL RESULTS

RAW MATERIAL CHARACTERIZATION STUDIES

Mineralogy

Mineral content of the augered glacial lake clays and some commercial samples were estimated using the results from X-ray diffraction data, chemical analyses, thermogravimetric analyses (TGA), and differential thermal analyses (DTA).

X-Ray Analyses

In the initial X-ray diffraction studies, manual comparisons of integrated intensity peaks of the bulk samples were made. Mineral phases for the bulk-augered and selected commercial samples are shown in tables 6 and 7, respectively; the relative peak intensity variations from sample to sample do not necessarily indicate quantitative mineral composition changes.[6]

The glacial lake clays were found to contain only small quantities of the clay minerals such as kaolinite, chlorite, and smectite. They also contained major to minor amounts of quartz, plagioclase, microcline, calcite, dolomite, and mica derived from igneous and metamorphic rocks abraded by the glaciers. These nonclay minerals were found in sand size (50 to 2,000 μm), silt size (2 to 50 μm) and some in clay size (<2 μm), as reported previously (5-6). Commercial western bentonite samples contained smectite, small quantities of quartz, cristobalite, feldspar, calcite, dolomite, mica, and kaolinite. X-ray data of the bulk sample were most useful for determining the impurities in the clay samples but not very useful for determining the clay content.

To better quantify the smectite content of augered glacial lake clays, a few bulk samples were subjected to the sedimentation-glycolation method (11) used at the University of Minnesota to produce clay-size fractions. These fractions were determined by gravity settling, centrifuging, and drying the centrifuge suspension (table 8). The fine fractions contained more smectite than did the coarse fractions. The combined fine and coarse fractions from sample VI-37A contained the most smectite (34-50 pct) of the Minnesota samples, but this smectite content was still less than the >50 pct contained in the typical commercial western bentonite (WB-17).

[6]Relative integrated intensities are affected by several factors not corrected for here and therefore these results must be used with caution. Values given may be compared from sample to sample (vertically) as a clue to greater or lesser amounts of that phase, but cannot be used to determine relative abundance of the various phases within one sample (horizontally).

TABLE 6. - Relative integrated intensities of major X-ray diffraction peaks[1] for minerals in augered glacial lake clay and shovel samples

Sample	Auger section, ft	Quartz	Calcite	Dolomite	Plagioclase	Microcline	Mica	Chlorite	Kaolinite
Area I:									
1A.....	10-15	2,601	1,063	1,608	369	ND	23	45	26
1B.....	25-30	3,493	428	361	529	566	52	90	132
1C.....	45-50	5,506	912	1,190	740	475	52	66	119
1D.....	70-75	4,651	697	1,024	1,056	615	52	4	100
2A.....	5-10	2,153	605	350	428	441	40	34	88
2B.....	25-30	3,411	424	778	635	ND	ND	61	98
2C.....	70-75	3,600	502	778	600	ND	38	ND	100
3A.....	15-20	5,329	605	973	396	1,176	44	45	42
3B.....	45-50	4,597	876	1,296	630	361	18	ND	96
3C.....	70-75	4,122	630	864	630	384	48	31	108
4A.....	0- 5	2,905	724	454	484	335	ND	77	81
4B.....	20-25	1,910	388	299	400	331	34	17	94
4C.....	35-40	4,356	697	1,537	625	250	37	ND	92
4D.....	70-75	4,462	538	630	566	269	27	ND	74
5A.....	5-10	2,938	595	740	437	292	38	61	222
5B.....	30-35	2,830	384	416	400	276	36	53	119
5C.....	50-55	5,991	506	635	620	1,714	24	34	76
7A.....	15-20	1,815	640	346	372	276	18	55	88
7B.....	40-45	5,300	1,069	2,714	666	ND	ND	ND	62
7C.....	60-65	2,228	493	400	408	303	42	52	132
7D.....	72-75	3,684	342	428	471	231	58	85	146
Area II:									
32A.....	5-10	6,320	328	1,069	1,211	671	69	69	104
32B.....	30-35	7,885	718	2,421	1,109	506	52	34	132
32C.....	45-50	4,186	1,122	1,376	548	286	21	64	ND
33A.....	15-20	6,480	462	1,592	1,253	480	77	96	144
33B.....	30-35	5,535	751	1,640	734	253	35	44	154
33C.....	45-50	3,894	1,232	1,544	595	ND	41	61	128
Area III:									
9A.....	8-10	8,724	502	1,391	3,014	835	34	28	72
9B.....	25-30	4,610	282	645	1,289	20	71	144	213
10A.....	10-15	8,798	655	1,089	2,352	1,018	52	27	86
10B.....	15-20	7,974	392	942	2,500	1,197	108	56	100
11A.....	10-15	4,956	306	640	1,239	790	35	151	125
12A.....	15-20	5,944	279	272	1,815	1,076	42	18	156
Area IV:									
13A.....	15-20	3,014	980	967	835	388	49	135	144
13B.....	30-35	1,910	1,149	640	475	ND	49	19	100
14C.....	5-10	3,745	949	847	918	458	55	114	142
14D.....	30-35	1,918	1,204	666	552	ND	48	74	123
15A.....	5-10	2,500	1,429	888	681	292	22	98	117
15B.....	20-25	2,520	1,303	1,011	543	234	ND	121	102
15C.....	45-50	2,570	1,279	930	955	350	ND	106	149
16A.....	5-10	3,091	1,544	1,163	924	372	ND	37	110
16B.....	20-25	2,381	936	729	640	ND	41	67	81
16C.....	40-45	2,401	1,149	784	681	196	ND	77	108
17A.....	5-10	4,858	620	650	1,866	586	ND	79	108
17B.....	20-25	4,303	71	213	2,788	936	32	ND	38

See explanatory notes at end of table.

TABLE 6. - Relative integrated intensities of major X-ray diffraction peaks[1] for minerals in augered glacial lake clay and shovel samples--Continued

Sample	Auger section, ft	Quartz	Calcite	Dolomite	Plagioclase	Microcline	Mica	Chlorite	Kaolinite
Area V:									
18A.....	10-15	4,597	1,170	2,007	1,282	ND	24	22	46
18B.....	20-25	4,173	1,354	2,520	1,289	324	24	ND	49
18C.....	30-35	5,700	1,376	2,440	1,406	458	53	26	41
19A.....	5-10	5,184	1,354	2,285	2,209	437	ND	26	56
19B.....	20-25	5,402	1,318	2,181	3,832	1,537	61	ND	42
19C.....	30-35	6,691	1,498	2,756	1,116	534	28	ND	52
20A.....	5-10	5,127	1,452	2,411	1,945	520	38	8	48
20B.....	15-20	4,422	1,376	2,663	1,498	807	ND	42	56
20C.....	25-30	5,883	1,170	2,440	2,228	1,082	55	ND	52
21A.....	5-10	5,358	1,459	2,256	2,520	524	ND	55	44
21B.....	45-50	5,700	1,498	2,440	762	566	18	58	52
22A.....	5-10	5,975	1,253	2,275	1,239	949	45	ND	56
22B.....	15-20	6,448	1,347	2,362	2,333	630	46	ND	40
22C.....	40-45	5,550	1,225	2,777	1,436	511	53	ND	62
23A.....	10-15	6,545	1,190	2,820	1,142	906	46	ND	58
23B.....	25-30	6,384	1,176	2,777	2,052	372	71	4	ND
23C.....	40-45	5,670	1,274	2,450	1,340	502	ND	ND	61
25A.....	25-30	5,791	1,050	2,294	1,739	888	48	26	ND
Area VI:									
36A.....	15-20	2,927	156	493	174	ND	12	100	94
36B.....	30-35	2,098	328	506	296	ND	15	44	96
36C.....	70-75	3,036	543	756	250	ND	49	ND	100
37A.....	25-30	2,981	185	1,050	328	182	31	69	128
37B.....	45-50	2,490	335	620	240	256	31	119	96
37C.....	70-75	3,102	576	980	361	234	64	77	98
Area VII:									
38A.....	5-10	2,181	740	640	635	282	ND	ND	76
38B.....	25-30	1,858	894	571	586	250	ND	ND	106
38C.....	45-50	1,648	942	595	441	172	ND	ND	74
Area VIII:									
39A.....	15-20	2,788	936	888	625	ND	ND	ND	85
39B.....	30-35	3,721	778	734	762	286	ND	ND	76
39C.....	45-50	1,980	824	697	586	ND	ND	ND	58
Area IX:									
40A.....	15-20	3,091	1,096	1,823	412	388	ND	ND	ND
40B.....	45-50	3,249	876	1,656	480	228	ND	ND	ND
41A.....	15-20	3,684	ND	2,520	784	ND	ND	ND	ND
41B.....	45-50	12,432	ND	1,866	1,030	299	ND	ND	ND
BRN-43.....	NE	11,990	ND	ND	ND	ND	ND	ND	ND
WLH-52.....	NE	2,181	380	462	174	ND	ND	ND	ND
ITS-46[2].....	NE	49	ND	ND	ND	ND	ND	ND	21
STC-53.....	NE	11,925	130	204	1,056	ND	72	ND	86

NE Not evaluated.

ND None detected.

[1]See text footnote 6.

[2]Peak heights of geothite and hematite were 280 and 290, respectively.

TABLE 7. - Relative integrated intensities of major X-ray diffraction peaks[1] for minerals in commercial samples

Sample	Quartz	Calcite	Dolomite	Plagioclase	Microcline	Mica	Chlorite	Kaolinite	Cristobalite
KA-1.....	1,858	ND	ND	ND	ND	ND	ND	3,457	ND
SB.....	1,498	ND	ND	46	ND	40	ND	ND	ND
WB-3.....	276	303	44	213	ND	ND	ND	ND	1,253
WB-4.....	276	108	169	615	237	59	ND	ND	1,513
WB-5.....	538	188	123	454	671	31	ND	ND	1,452
WB-6.....	955	ND	ND	296	ND	ND	ND	ND	96
WB-8.....	475	620	ND	ND	ND	ND	ND	12	ND
WB-9.....	2,052	269	ND	ND	ND	174	ND	ND	ND
WB-10.....	2,830	339	ND	538	ND	66	ND	ND	ND
WB-11.....	85	ND	36	462	44	56	ND	ND	1,624
WB-12.....	2,916	111	ND	1,136	462	77	ND	ND	ND
WB-13.....	1,274	135	ND	615	292	ND	ND	ND	ND
WB-14.....	1,116	ND	ND	778	376	262	ND	40	ND
WB-16.....	289	ND	49	346	ND	ND	ND	ND	56
WB-17.....	1,475	117	72	918	286	ND	ND	ND	199

ND None detected. [1]See text footnote 6.

TABLE 8. - X-ray diffraction analysis[1] of fractions of selected clay and bentonite samples

Sample	Clay[2]		Relative mineral abundance[3]									Interstratification
	Fraction	pct	SM	VE	CH	I	KA	QU	FE	CA	D	
I-7B.....	Fine....	6	5	ND	ND	1	3	1	ND	ND	ND	I-VE = 4, I-SM = 4
I-7B.....	Coarse..	8	3	ND	2	2	2	2	2	ND	2	I-VE = 4, I-SM = 4
II-32C....	Fine....	15	4	ND	1	3	1	ND	2	1	ND	K-SM = 2, I-SM = 4
II-32C....	Coarse..	16	3	ND	2	2	2	1	2	3	ND	I-VE = 4, I-SM = 4
III-9B....	Fine....	6	4	ND		2	2	1	2	ND	ND	K-SM = 3, I-SM = 5
III-9B....	Coarse..	6	2	ND	2	3	3	2	4	ND	ND	I-VE = 2, I-SM = 2
IV-15B....	Fine....	2	2	ND	1	2	2	1	2	ND	ND	K-SM = 3, I-SM = 5
IV-15B....	Coarse..	14	2	ND	2	2	3	2	4	2	ND	I-VE = 3, I-SM = 3
V-23A.....	Fine....	<1	4	ND	ND	2	3	1	2	1	ND	K-SM = 2, I-SM = 4
V-23A.....	Coarse..	27	2	1	1	2	2	2	2	3	3	I-VE = 3, I-SM = 3
VI-37A....	Combined	74	5	1	1	3	3	2	3	ND	2	ND
VII-38A...	..do....	64	3	3	2	2	2	2	3	3	2	I-VE = 2
VIII-39A..	..do....	46	2	4	1	2	2	2	3	4	2	I-VE = 1
IX-40A....	..do....	37	2	5	1	2	1	1	2	3	2	I-VE = 1
IX-41A....	..do....	21	4	4	2	3	2	2	3	ND	ND	ND
WB-17.....	..do....	88	6	ND	ND	ND	ND	1	1	ND	ND	ND

CA Calcite. FE Feldspar. ND None detected. SM Smectite.
CH Chlorite. I Illite. QU Quartz. VE Vermiculite.
D Dolomite. KA Kaolinite.

[1]Conducted by University of MN Soils Survey Laboratory.

[2]Combined, coarse, and fine refer to <2-, 2- to 0.2-, and <0.2-μm particle sizes, respectively; percentages based on initial sample weight.

[3]1, trace (<5 pct); 2, very small (5-10 pct); 3, small (11-20 pct); 4, moderate (21-33 pct); 5, abundant (34-50 pct); 6, dominant (>50 pct).

Chemistry

Chemical analyses were conducted as a means to help delineate the types of minerals present in clays. The aluminum and silicon elemental analyses of the commercial bentonites (table 5) were similar to the midwest clays (tables 3-4), except for sample ITS-46, which had much lower values. This material, paint rock, is a highly altered part of the slaty iron formation on the Mesabi Range. Alteration by weathering destroyed most of all the former quartz, iron silicates, and iron carbonate, leaving a clay-textured material high in aluminum silicates and iron oxides. Completely altered paint rock contains mostly hematite, quartz, and kaolinite and small quantities of a smectite (the iron-bearing nontronite). The claylike texture, high iron oxide-low silica content, and availability on the Iron Range prompted the testing of this sample as a binder for agglomerating iron ore concentrate.

Because bentonite contains mainly smectite (aluminum silicate mineral), the Si-Al ratio of the samples was evaluated to estimate the relative smectite content. Glacial lake clays, however, contained only a little smectite and various quantities of four or five other aluminum silicate minerals. Therefore, the Si-Al ratio was not a useful criterion for estimating the smectite content in glacial lake clays.

Thermal Analyses

Thermogravimetric and differential thermal analyses were also used for the determination of poorly crystalized complex minerals. With these methods, a sample of about 0.05 g was tested in a Perkin-Elmer thermoanalyzer at a heating rate of about 25° C/min. The thermogravimetric analysis (TGA) results (fig. 2) indicated that the greatest weight change started to occur at about 400° C for illite (I-S) and the clay sample

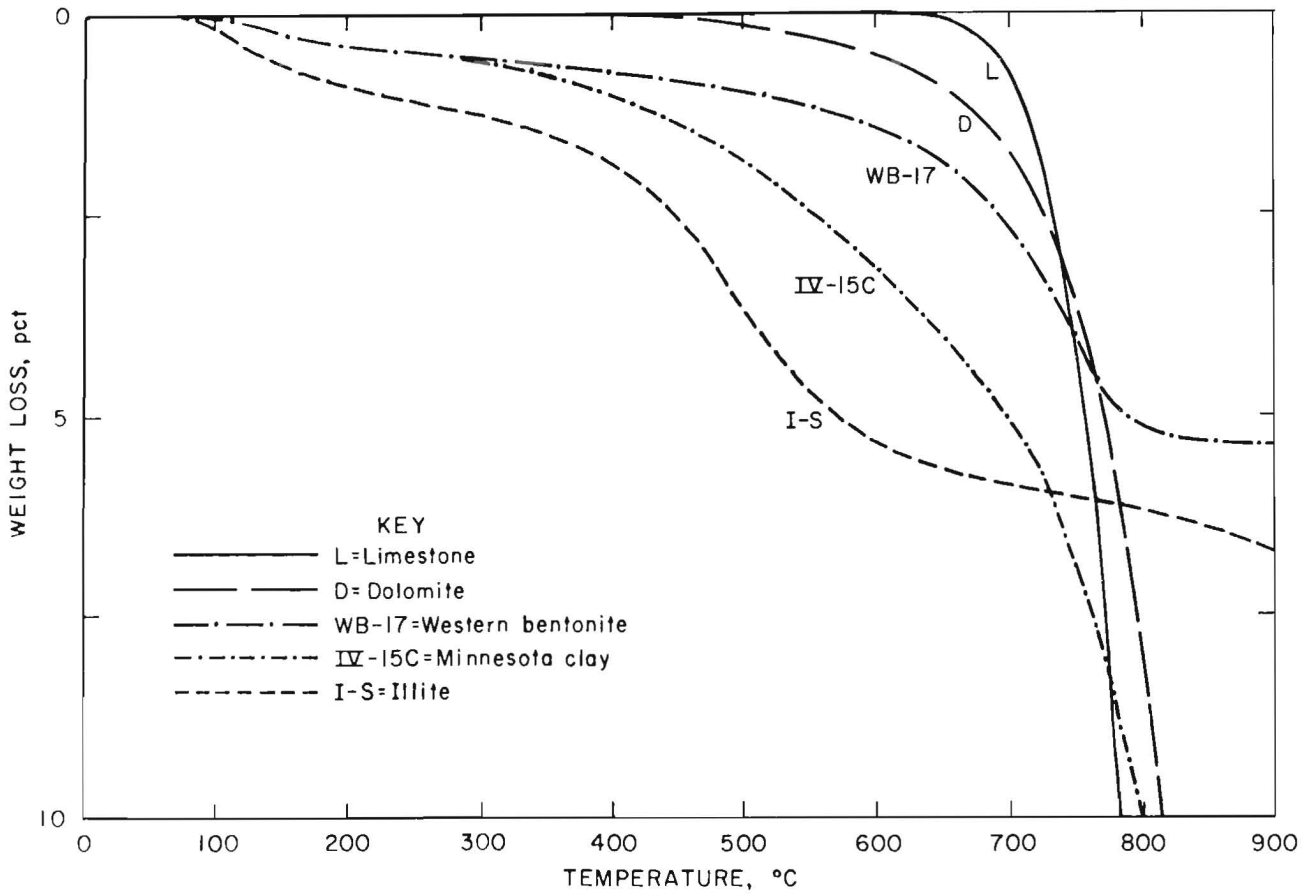

FIGURE 2.—Thermogravimetric analysis of selected samples.

IV-15C and at about 700° C for a western bentonite (WB-17), dolomite (D), and limestone (L). Dolomite and limestone samples contained principally calcium magnesium carbonate and calcium carbonate, respectively.

The TGA data were more meaningful when they were used in conjunction with the differential thermal analysis (DTA) results shown in figure 3. With all clay samples, the TGA curves showed a gradual weight loss but the DTA curves showed narrow temperature ranges of exothermic and endothermic peaks. An exothermic peak obtained with the clay sample VI-37A between 350° and 450° C was probably because of the presence of organic materials. Dolomite, chlorite, and Minnesota clay samples had exothermic peaks at about 900° C. A strong endothermic peak, observed in all the samples between 100° and 300° C, was probably because of the removal of hygroscopic, interlayer and/or coordinated water. At 600° C, the kaolinite (KA-1) sample had a strong endothermic peak, which is in agreement with the results reported by Hoffman (12). Southern bentonite yielded only a small peak at about 700° C, while western bentonite (WB-17) produced a large endothermic peak between 700° and 800° C. The Minnesota clay sample had a small endothermic peak at 600° and 800° C, which suggested that only small quantities of kaolinite, smectite, and limestone were present.

Swelling-Shrinkage and Clay Briquet Strengths

Three types of swelling-shrinkage procedures were used to characterize the inherent properties of the clay that ultimately could be related to the iron ore pellet strengths. One procedure involved the evaluation of the briquet shrinkage and strength, and the other two procedures involved the swelling of dry clay powder in water as described in appendix A.

The briquet procedure consisted of pressing a clay paste into 27 holes (1/2-in diam) drilled through a 5/8-in-thick Teflon (fluorocarbon polymer) plate. About 20 pct water was added to the dried

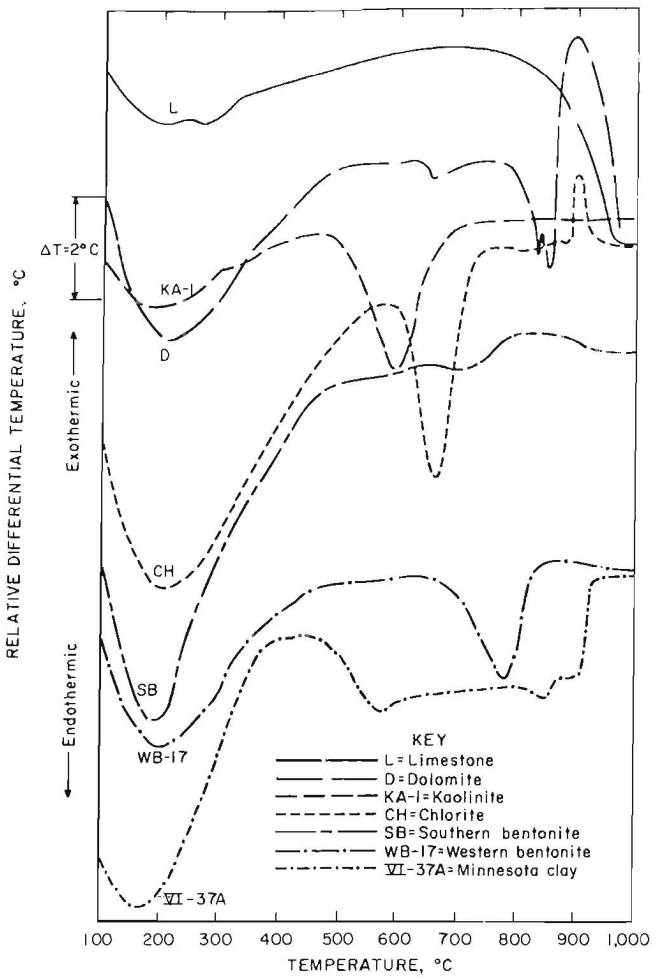

FIGURE 3.—Influence of temperature on endothermic and exothermic behavior of different samples.

Minnesota clay powder while about 40 pct water had to be added to the western bentonites to make pastes of about the same consistency. The filled sample plate was dried overnight at 75° C. The briquet volumetric shrinkages were generally less than 20 pct with the Minnesota clays (table 2), and were over 20 pct with the western bentonites. The compressive strengths of the dry clay briquets were less than 255 lb with the Minnesota clays and more than 275 lb with the western bentonites. Even though this method revealed a difference between Minnesota clays and western bentonites, the crudeness of the test and the small magnitude of these differences make this test imprecise for clay evaluation.

Two swelling procedures were used to evaluate the samples. The first swelling procedure was the dilatancy (DIL) method,

which involved adding dry clay powder slowly to water and then determining the ratio of the final wet (V_f) and initial dry (V_i) volumes as described in appendix A. With the Minnesota clay samples, these ratios were less than 3 (table 9), but with the western bentonites (WB's) dried below 500° C, ratios over 5 (table 10) were obtained. With the augered Minnesota samples, the highest ratios were obtained with the Red River Valley samples.

A second swelling method (PWAT-V) was evaluated in conjunction with the PWAT number weight gain experiments as described in appendix A. Both swelling methods produced similar results (tables 9 and 10), but more reproducible data were obtained with the dilatancy method. However, the dilatancy method required considerably more time for conducting the measurements.

Particle Size

Particle size is usually an important variable in characterizing clay-bearing materials because one of the definitions of clay is "a 2-μm-diam or smaller particle." However, nonclay minerals also occur in this size range and are referred to as rock flour; rock flour can be produced by the grinding action of a glacier. The particle size distributions of the clay samples were determined by optical laser and sedimentation methods. With the optical laser method, the mass median particle sizes for some selected augered Minnesota samples ranged from 6 to 13 μm, while particle sizes for western bentonites ranged from 4 to 6 μm. These results indicated very little difference in the mean particle size between the augered Minnesota samples and western bentonites.

The laser method only measured particles larger than 2 μm. Therefore, further tests were conducted with the U.S. Department of Agriculture (USDA) sedimentation method (13), which was used to measure the particle sizes down to 0.2 μm (fig. 4). One reason that smaller apparent mean sizes were obtained with the sedimentation method was probably because of the low density of the expanded clay particles, which resulted in slow settling rates. The typical western bentonite (WB-17) had the highest percentage of minus 0.2-μm particles; the next highest were the Red River Valley clay samples (VI-36B and VI-37A).

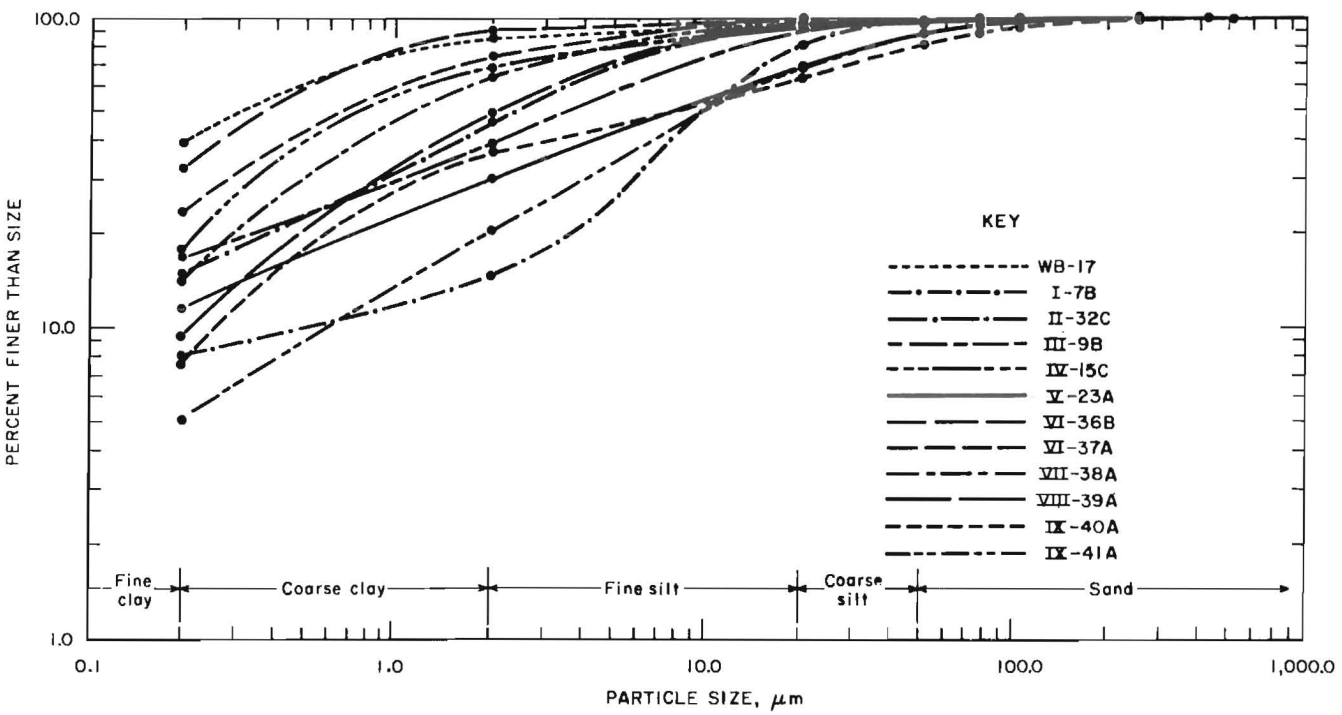

FIGURE 4.—Particle size distribution of different samples with the sedimentation method.

TABLE 9. - Binder characterization data of Minnesota samples

Sample	Auger section, ft	CEC,[1] meq/100 g		Electrode values[2]				Colloid, pct		Swell,[3] (V_f/V_i)		PWAT No.[4]	η,[5] cP
		mb	BTAC-Na	Na^+	Ca^{2+}	Eh	pH	Total	Fine	DIL	PWAT-V		
Area I:													
1A.....	10-15	15	NE	8.5	18.5	147	7.8	7.1	1.1	NE	0.76	78	NE
1B.....	25-30	15	NE	8.4	18.8	150	7.8	6.6	.6	NE	.21	85	NE
1C.....	45-50	10	NE	8.1	20.2	148	7.6	6.5	2.0	0.95	.64	40	NE
1D.....	70-75	15	NE	8.1	19.4	148	7.5	8.2	2.0	NE	.61	44	6.5
2A.....	5-10	15	NE	9.1	15.1	144	7.6	5.3	.6	NE	.47	68	NE
2B.....	25-30	15	NE	8.3	20.0	141	7.7	5.2	1.4	NE	.50	48	NE
2C.....	70-75	15	NE	8.1	21.9	146	7.3	4.9	1.7	NE	.37	56	NE
3A.....	15-20	15	NE	8.3	18.1	140	7.9	8.0	1.1	NE	.42	58	NE
3B.....	45-50	15	NE	8.5	23.0	144	7.6	5.3	1.7	NE	.50	54	NE
3C.....	70-75	15	NE	8.5	33.9	147	7.6	4.9	.3	NE	.50	65	NE
4A.....	0- 5	15	NE	7.6	21.6	136	7.8	8.3	.8	NE	.72	99	NE
4B.....	20-25	15	NE	8.2	14.6	140	7.8	5.3	NE	NE	.70	94	NE
4C.....	35-40	10	NE	NE	NE	NE	NE	NE	NE	NE	.61	57	NE
4D.....	70-75	15	NE	NE	NE	NE	NE	NE	NE	NE	.64	72	NE
5A.....	5-10	15	NE	12.7	72.7	199	8.3	5.6	NE	NE	.51	63	NE
5B.....	30-35	15	NE	8.8	19.1	138	7.8	NE	NE	NE	.61	85	NE
5C.....	50-55	10	NE	8.7	20.8	138	7.7	NE	1.1	NE	.36	52	NE
7A.....	15-20	15	NE	10.4	11.8	124	7.7	NE	NE	NE	.61	100	NE
7B.....	40-45	15	0.4	9.2	23.4	135	7.8	13.8	5.6	1.16	.33	30	9.5
7C.....	60-65	15	NE	2.2	15.1	265	8.4	12.2	6.6	NE	NE	132	NE
7D.....	72-75	10	NE	1.7	12.8	235	8.5	14.1	4.6	NE	NE	106	NE
Area II:													
32A.....	5-10	15	NE	.7	21.2	245	8.1	23.1	14.8	NE	NE	69	NE
32B.....	30-35	5	NE	.7	14.7	227	8.3	11.9	5.6	NE	NE	NE	NE
32C.....	45-50	15	.6	NE	NE	NE	NE	31.1	NE	1.97	.83	95	6.5
33A.....	15-20	10	NE	4.0	14.8	210	8.6	27.6	11.7	NE	NE	34	NE
33B.....	30-35	10	NE	1.1	18.3	212	8.3	19.3	8.7	.92	NE	40	8.5
33C.....	45-50	15	NE	1.2	25.0	210	8.2	29.3	18.7	NE	NE	105	NE
Area III:													
8A.....	15-20	10	NE	.9	12.4	202	8.4	28.5	7.7	NE	NE	50	NE
9A.....	8-10	5	NE	.6	12.0	208	8.3	19.1	2.3	NE	NE	26	NE
9B.....	25-30	10	.5	1.1	11.4	206	8.3	11.7	5.9	.60	.47	41	6.5
10A.....	10-15	10	NE	NE	NE	NE	NE	NE	NE	NE	NE	7	NE
10B.....	15-20	10	NE	.6	12.9	203	8.4	26.8	9.7	1.27	NE	17	NE
11A.....	10-15	10	NE	.6	9.9	201	8.3	21.4	4.9	NE	NE	37	NE
12A.....	15-20	10	NE	.9	10.2	198	8.4	20.1	5.9	NE	NE	14	NE
Area IV:													
13A.....	15-20	15	NE	.9	15.2	199	8.3	24.6	17.8	NE	NE	50	NE
13B.....	30-35	15	NE	1.3	15.2	195	8.3	22.8	7.7	NE	NE	51	NE
14A.....	5-10	15	NE	.6	14.4	278	8.3	5.0	.8	NE	NE	70	NE
14B.....	30-35	15	NE	1.4	15.6	223	8.1	8.7	2.0	1.17	NE	92	NE
15A.....	5-10	15	NE	1.1	14.8	304	8.2	4.7	1.4	NE	NE	78	NE
15B.....	20-25	20	NE	1.5	14.8	275	8.3	2.9	.5	NE	NE	84	NE
15C.....	45-50	15	NE	1.9	17.1	245	8.2	5.6	.1	1.10	.78	101	6.0
16A.....	5-10	15	.6	.8	14.1	266	8.3	4.3	1.7	NE	NE	63	NE
16B.....	20-25	15	NE	1.4	14.5	232	8.2	5.9	1.9	NE	NE	88	NE
16C.....	40-45	15	NE	7.0	47.6	218	7.2	16.0	1.6	NE	NE	NE	NE
17A.....	5-10	15	NE	.7	15.9	280	8.2	1.4	2.3	NE	.75	79	NE
17B.....	20-25	5	NE	NE	NE	NE	NE	NE	NE	NE	NE	30	NE

See explanatory notes at end of table.

TABLE 9. - Binder characterization data of Minnesota samples--Continued

Sample	Auger section, ft	CEC,[1] meq/100 g		Electrode values[2]				Colloid, pct		Swell,[3] (V_f/V_i)		PWAT No.[4]	η,[5] cP
		mb	BTAC-Na	Na^+	Ca^{2+}	Eh	pH	Total	Fine	DIL	PWAT-V		
Area V:													
18A.....	10-15	10	NE	1.1	12.6	274	7.8	16.6	0.2	NE	NE	46	NE
18B.....	20-25	15	NE	1.3	14.6	269	7.8	13.3	.1	1.10	NE	43	NE
18C.....	30-35	15	NE	.9	15.6	267	7.9	24.1	.2	NE	NE	40	7.0
19A.....	5-10	15	NE	1.0	13.1	264	7.9	21.3	.1	NE	NE	31	NE
19B.....	20-25	15	NE	.9	15.5	265	7.9	30.3	NE	1.45	NE	41	NE
19C.....	30-35	10	NE	1.3	16.8	258	7.8	16.9	.1	NE	NE	41	NE
20A.....	5-10	10	NE	1.2	13.8	276	7.7	10.7	.1	NE	NE	NE	NE
20B.....	15-20	10	NE	NE	NE	NE	NE	NE	NE	NE	0.76	68	NE
20C.....	25-30	10	NE	NE	NE	NE	NE	NE	NE	NE	NE	50	NE
21A.....	5-10	15	NE	NE	NE	NE	NE	NE	NE	NE	NE	38	NE
21B.....	45-50	5	NE	NE	NE	NE	NE	NE	NE	NE	NE	39	NE
22A.....	5-10	15	NE	NE	NE	NE	NE	NE	NE	NE	.79	44	NE
22B.....	15-20	15	NE	.9	13.3	259	7.8	18.9	.4	NE	NE	40	NE
22C.....	40-45	10	NE	NE	NE	NE	NE	NE	NE	NE	.82	54	NE
23A.....	10-15	10	0.1	.9	16.8	260	7.9	26.9	.1	1.72	1.25	72	6.75
23B.....	25-30	10	NE	1.0	16.7	259	7.9	29.2	.3	NE	.87	54	NE
23C.....	40-45	10	NE	1.3	17.3	261	7.9	18.2	.1	NE	.82	60	NE
25A.....	25-30	15	NE	1.1	17.8	265	7.8	15.0	<.1	NE	.76	57	NE
Area VI:													
36A.....	15-20	20	.9	6.0	79.0	112	7.7	2.3	NE	NE	1.38	106	NE
36B.....	30-35	25	1.1	6.2	22.3	119	7.7	8.2	NE	2.94	1.54	110	8.0
36C.....	70-75	20	.7	3.9	23.1	111	7.9	29.9	NE	2.50	1.32	107	NE
37A.....	25-30	20	1.0	4.8	16.7	101	7.8	5.3	NE	2.41	1.08	92	7.0
37B.....	45-50	20	.9	2.1	21.3	100	7.9	5.6	NE	2.26	NE	104	NE
37C.....	70-75	20	.7	1.8	19.5	129	7.8	35.0	NE	2.12	1.23	102	NE
Area VII:													
38A.....	5-10	15	<.1	8.0	7.2	143	8.2	36.9	2.5	NE	.65	90	NE
38B.....	25-30	15	<.1	3.8	13.0	157	8.2	32.9	1.0	NE	.61	93	NE
38C.....	45-50	20	<.1	3.5	12.4	156	8.2	21.5	.5	NE	.76	109	NE
Area VIII:													
39A.....	15-20	10	.1	3.1	11.6	153	8.2	27.9	.5	NE	.64	73	NE
39B.....	30-35	10	.2	3.0	8.4	151	8.2	26.0	.6	NE	.69	73	NE
39C.....	45-50	15	<.1	3.1	12.9	8	8.1	25.0	.8	NE	.74	98	NE
Area IX:													
40A.....	15-20	10	<.1	1.9	15.0	8	8.2	23.2	1.7	NE	.75	50	NE
40B.....	45-50	10	<.1	2.1	15.8	8	8.3	23.6	1.1	NE	.78	56	NE
41A.....	15-20	10	<.1	1.7	9.5	8	8.2	13.9	1.8	NE	.71	34	NE
41B.....	45-50	10	<.1	1.9	15.9	8	8.2	24.4	.9	NE	.72	50	NE
ITS-46....	NE	5	2.8	NE	NE	NE	NE	24.9	3.2	1.88	1.00	70	10.0

CEC Cation exchange capacity. DIL Dilatancy. NE Not evaluated.

[1]CEC determined by two methods: mb, methylene blue and BTAC, benzyltrimethyl ammonium chloride.

[2]Na^+ and Ca^{2+} (ppm) determined by selective ion electrodes; Eh (mV) determined with platinum electrode vs standard normal calomel electrode.

[3]V_f and V_i refer to final wet and initial dry sample volume, respectively; PWAT-V values determined from same sample as PWAT No. test.

[4]Determined by multiplying difference between wet and dry weight (ΔW) by 100 and dividing by initial weight (W_i).

[5]Viscosity (η) determined at 600 rpm.

TABLE 10. - Binder characterization data for commercial samples

Sample	CEC,[1] meq/100 g					Electrode[2] values				Colloid, pct		Swell,[3] (V_f/V_i)		PWAT No.[4]	η,[5] cP
	mb	BTAC				Na^+	Ca^{2+}	Eh	pH						
		Na^+	Ca^{2+}	K^+	Mg^{2+}					Total	Fine	DIL	PWAT-V		
CH.....	5	0.3	0.2	0.00	0.6	2.5	1.2	96	7.3	3.7	0.0	1.25	0.65	33	9
M-B....	10	NE	NE	NE	NE	1.9	16.9	91	7.9	29.6	20.3	1.49	.84	48	8
SG.....	5	3.4	.3	.10	.1	32.9	1.7	127	6.6	12.3	1.6	.76	.59	67	7
Z.....	NE	NE	NE	NE	NE	47.4	.3	117	9.2	11.9	2.2	NE	.72	50	9
KA-1...	10	1.6	.9	.03	.2	12.3	1.5	191	6.3	75.5	45.2	.58	.44	54	5
VG.....	5	.2	2.1	.10	2.0	2.3	10.5	113	7.4	11.9	.4	1.67	.90	21	8
KA-2...	10	.9	4.2	.30	2.5	1.1	15.7	274	4.1	5.9	.0	2.38	1.02	30	8
AT.....	15	.5	8.2	.30	4.1	1.5	11.0	94	7.8	83.6	76.6	1.16	.75	259	26
I-S....	15	.7	8.6	.60	1.1	1.2	9.2	154	6.7	20.0	9.6	.77	1.06	55	8
Al-C...	5	NE	NE	NE	NE	NE	NE	273	3.6	88.6	2.1	2.21	2.95	480	8
BE.....	25	19.1	11.6	.80	2.1	31.7	4.9	103	7.6	35.6	5.6	1.92	2.05	109	7
SB.....	40	1.2	41.0	.70	10.8	24.2	7.3	50	9.2	23.1	10.8	1.80	1.36	122	8
B-M....	20	3.0	.5	.00	2.0	28.5	.8	107	6.3	21.4	3.5	1.70	1.75	264	8
WB-3...	40	40.8	9.6	.30	2.5	292.0	40.7	78	8.9	74.3	17.3	5.66	3.15	518	20
WB-4...	40	24.6	7.2	.50	3.6	136.0	26.6	137	8.3	73.0	26.4	5.90	3.75	542	20
WB-5...	40	21.1	11.0	.30	6.6	NE	NE	NE	NE	79.5	13.4	10.20	4.71	575	100
WB-6...	45	41.7	11.6	1.10	3.1	279.0	22.2	89	8.9	78.4	10.2	15.00	6.21	811	43
WB-7...	40	49.5	1.0	.50	1.0	440.0	25.3	56	9.4	64.1	16.7	10.28	5.56	756	33
WB-8...	55	41.7	10.6	1.30	4.4	192.0	20.7	62	8.7	85.4	30.7	16.17	7.73	811	97
WB-9...	45	45.2	4.2	.40	.5	293.9	32.7	98	9.1	70.4	18.7	11.30	6.21	614	104
WB-10..	40	38.5	4.1	.40	.7	124.0	31.8	41	9.2	71.4	20.3	7.81	5.69	720	33
WB-11..	50	14.7	14.6	.30	14.3	19.3	72.4	181	7.1	87.8	36.0	9.70	3.12	540	28
WB-12..	45	49.8	3.3	.40	3.3	466.0	38.5	35	9.5	96.3	34.4	20.00	6.35	852	>150
WB-13..	50	36.8	8.4	1.30	2.3	297.0	40.9	40	9.3	79.6	31.0	15.59	7.50	1,004	82
WB-14..	50	37.6	9.8	1.30	3.4	NE	NE	NE	NE	94.7	39.5	16.40	6.36	786	48
WB-15..	50	37.6	4.2	.40	4.1	265.0	22.8	156	8.1	90.2	11.5	10.70	4.94	894	>150
WB-16..	55	34.7	17.6	.70	12.1	289.0	81.8	78	8.7	93.6	14.7	15.17	7.61	918	94
WB-17:.	55	32.4	12.9	1.00	5.9	247.0	46.0	37	8.2	82.0	29.5	19.60	6.90	930	88
200[6].	45	42.6	12.9	1.90	4.9	199.0	32.7	144	9.3	85.2	13.3	14.10	6.77	917	73
300[6].	50	40.0	12.9	1.90	4.4	NE	NE	NE	NE	NE	NE	NE	NE	NE	65
400[6].	45	42.6	12.9	2.00	4.4	161.0	28.8	143	9.2	75.9	11.1	12.20	6.73	864	49
500[6].	40	39.2	13.9	2.20	3.8	NE	NE	NE	NE	NE	NE	NE	5.84	693	NE
600[6].	35	40.9	14.9	2.40	4.1	133.0	12.8	158	8.5	48.9	7.0	8.75	5.00	612	9
700[6].	20	33.2	12.7	NE	4.8	100.0	1.9	172	8.2	8.1	1.0	3.69	2.12	200	11
800[6].	10	16.7	12.7	NE	4.4	68.1	NE	169	8.3	4.4	.3	1.36	1.04	58	13
WB-PA[7].	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	>150
ITS-GG[8]	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	242	>150

CEC Cation exchange capacity. DIL Dilatancy. NE Not evaluated.

[1]CEC determined by two methods: mb, methylene blue and BTAC, benzyltrimethyl ammonium chloride.

[2]Na^+ and Ca^{2+} (ppm) determined by selective ion electrodes; Eh (mV) determined with platinum electrodes vs standard normal calomel electrode.

[3]V_f and V_i refer to final wet and initial dry volume, respectively; PWAT-V values determined from same sample as PWAT No. test.

[4]Determined by multiplying difference between wet and dry weight (ΔW) by 100 and dividing by initial weight (W_i).

[5]Viscosity (η) determined at 600 rpm.

[6]Drying temperatures (°C); all other samples dried at 75° C.

[7]9 pct polyacrylate added to WB-17.

[8]17 pct GG-211D added to ITS-46.

A modified sedimentation technique was used to determine the total and fine colloid content. The total colloid content was defined as the quantity of material that remained in suspension after 18 h; a detailed description of this modified sedimentation procedure is given in appendix A. The total colloid contents of the augered samples (table 9) were less than 40 pct, compared with over 60 pct for the western bentonites dried at 75° C (table 10); the colloid content decreased as the bentonite drying temperature was increased above 200° C, which was probably because of the dehydration and densification of the sample. The 18-h suspension was allowed to settle for an additional 6 days, and then this suspension was centrifuged. The centrifuged sediment was defined as the fine colloid fraction. In general, the fine colloid content was lower with the Minnesota samples than with western bentonites.

A colloid particle settling rate is dependent on the ions in solution. Therefore the sodium, calcium, pH, and Eh (oxidation-reduction) electrode potential of the supernatant solutions from the colloid tests were determined as described in appendix A. The solutions from most of the western bentonite samples (table 10) contained over 100 ppm Na^+; while the solutions from the augered Minnesota samples dried at 75° C (table 9), contained less than 20 ppm Na^+. The principal mineral in western bentonite is sodium montmorillonite, and the solubility of the sodium in this mineral or the exchange of some other ion for this element may be the reason for the high solution sodium analyses. The Minnesota clays contained very little sodium montmorillonite and had lower dissolved sodium contents. The calcium ion analyses of the Minnesota clays were generally higher than the western bentonites. The pH values of the Minnesota clays were not significantly different from those obtained with the western bentonites.

Water Sorption

The degree of water sorption of the samples were determined by both the plate water absorption test (PWAT) and the adsorption method. The plate water absorption test consisted of measuring the 18-h weight gain of the sample after it was placed on top of a wet, porous ceramic plate that was immersed in distilled water to within 6 mm from the top of the plate, as described in appendix A. The Minnesota samples had relatively low PWAT numbers, i.e., less than 200 (table 9), as compared to 500 to 1,000 for western bentonites dried at 75° C (table 10); at drying temperatures above 200° C, the PWAT numbers of WB-17 began to decrease significantly.

The adsorption method involved placing the samples in a 100-pct relative humidity chamber for 18 h. Unlike the plate water absorption test, in this method the samples were not in direct contact with a wet surface. The weight adsorbed with the western bentonites ranged from 23 to 34 pct. These weight gains were smaller than those obtained by the direct water contact test (PWAT), which ranged between 500 and 1,000 pct. Silica gel, colloidal alumina, and zeolite gained 34, 20, and 8 pct by weight, respectively, with the adsorption method; with the plate water absorption test, values less than 100 pct weight gain were obtained, except for colloidal alumina, which had a value of 480 (table 10). In the plate water absorption test, the samples picked up water by both absorption and adsorption, with absorption being the major weight gain mechanism. It appears that the foliated platelike structure of the western bentonites absorbed large quantities of water by capillary action.

The PWAT numbers were also determined for selected commercial organic materials, both alone and mixed with clays. Although the ASTM PWAT method specifies the use of pure (distilled) water, these latter experiments also were conducted with Minneapolis, MN, tap water and wash water obtained from slurrying magnetic concentrate at 50 pct solids. The distilled, tap, and concentrate wash water contained respectively 4, 22, and 90 ppm Ca; 1, 2, and 76 ppm K; 1, 3, and 1,200 ppm Mg; and 1, 7, and 160 ppm Na. Plant waters from several pelletizing companies also were analyzed, and the concentrations of these cations were within the

range of the values obtained with the tap and concentrate wash water used in these tests. The results in table 11, show that for inorganic binders, western bentonite (WB-17) had the highest PWAT number with distilled water. However, the values dropped considerably with tap water or concentrate wash water. In general, with the guar gum organics, the PWAT numbers showed the least dependence on the type of water. Starch-graft copolymer (SGC), type J, had the highest PWAT number of all the binders, but it also decreased considerably as the ionic concentration of the water increased. The organic materials had higher PWAT numbers than did the Minnesota clays. When organic materials were added to the clays, the PWAT numbers increased proportionately to the quantity of organic material added. For example, with five parts paint rock (ITS-46) and three parts pregelatinized starch (PS-300), a PWAT number of 1,213 was obtained with distilled water.

These results indicated that the impurity level in the water influenced the PWAT results. The Minnesota clays had much lower PWAT numbers than the organic materials, but with a clay and organic mixture, higher PWAT numbers than with western bentonites were obtained (tables 9 and 10).

Cation Exchange Capacity

Two cation exchange capacity (CEC) methods were used for characterizing the clay samples. The methylene blue method (CEC-mb) involved titrating a clay slurry with a methylene blue dye solution until excess dye was observed (14) as described in appendix A. The CEC-mb values obtained with the western bentonites ranged from 40 to 55 meq/100 g (table 10); those for the augered Minnesota clays were 25 (or less) meq/100 g (table 9).

Because the sodium cation was suspected to be the most important constituent, a second CEC evaluation method was used to determine the individual cation exchange capacities. This alternative method (CEC-BTAC) involved measuring the sodium, potassium, calcium, and magnesium ions in solution after mixing benzyltrimethyl ammonium chloride (BTAC) with the clay slurry. In general, the sodium values with the western bentonites ranged from 20 to 50 meq/100 g (table 10), while with the Minnesota clay the sodium values were less than 3 meq/100 g (table 9). However, this method was not reliable when water-soluble, nonclay, sodium compounds were present. Soda ash, which sometimes is added to improve bentonite quality, caused erroneously high CEC-BTAC values. Poor correlations were observed among the potassium, calcium, and magnesium cation exchange values and the clay characteristics, such as colloid content, swelling, and PWAT.

Viscosity

Viscosity measurements were made with 6-pct clay slurries (15) as described in appendix A. The viscosities of western bentonites were greater than 20 cP, while the Minnesota clay viscosities were less than 10 cP (tables 9-10).

The viscosities of western bentonite (WB-17) were influenced by drying temperatures more than by any other clay characterization variable. For example, table 10 shows that the viscosity decreased from 88 to 9 cP when the drying temperature was increased from 75° to 600° C. Within this same temperature range, considerably less than a tenfold change was obtained with the other clay characteristics, such as, PWAT, swelling, colloid content, and cation exchange capacity. When the drying temperatures were increased from 75° to 200° C, the viscosity decreased by about 10 pct.

PELLETIZING STUDIES

The performance of the clay binders was evaluated by a batch pelletizing procedure, as described in appendixes B and C. Pelletizing tests were conducted with split-tube, shovel, auger, and commercial samples at up to 5-pct addition levels to the iron ore concentrate.

TABLE 11. - PWAT values and pellet properties obtained with different clays and organic mixtures

Binders	pct	PWAT No.[1] H_2O			Unfired				Fired strength, lb		R_{40},[2] pct/min		t_{90},[3] min		RDI,[4] pct		Porosity, pct
		Pure	Tap	Wash	H_2O, pct	Drop No.	WCS, lb	DCS, lb	5 1,200	5 1,250	5 1,200	5 1,250	5 1,200	5 1,250	Plus 6.3 mm	Minus 0.5 mm	
Organic:																	
None.....	0.00	NE	NE	NE	6.6	3.2	3.1	2.3	284	375	0.80	0.69	150	178	NE	NE	25.6
CMC-P.....	.05	NE	NE	NE	8.0	3.2	1.7	4.4	347	501	.75	.38	190	269	NE	NE	NE
	.10	NE	NE	NE	8.0	6.9	3.1	8.5	254	347	.71	NE	178	NE	91.3	7.2	NE
	100.00	2,550	2,060	1,800	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
CMC-H.....	.10	NE	NE	NE	9.0	7.0	4.1	7.7	173	673	.76	.50	183	216	90.9	8.8	NE
	100.00	3,260	3,070	1,990	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
SGC-A.....	.50	NE	NE	NE	12.7	5.8	1.0	1.0	63	262	NE	NE	NE	NE	NE	NE	NE
	100.00	14,000	3,700	5,390	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
SGC-J.....	.20	NE	NE	NE	9.2	5.0	2.8	3.7	204	NE	NE	NE	NE	NE	NE	NE	NE
	100.00	42,000	NE	9,500	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
PS-200.....	.10	NE	NE	NE	6.6	4.4	3.3	11.3	444	NE	.57	NE	215	NE	89.1	3.5	23.6
	.30	NE	NE	NE	7.7	10.4	5.2	41.8	274	647	.75	NE	170	NE	90.9	3.6	NE
	100.00	870	785	750	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
PS-300.....	.30	NE	NE	NE	7.3	4.4	2.6	19.6	289	769	NE	NE	NE	NE	86.4	5.6	NE
	100.00	3,530	2,680	1,000	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
GG-J.....	.05	NE	NE	NE	9.4	9.6	4.2	5.1	273	NE	NE	NE	NE	NE	NE	NE	NE
	.10	NE	NE	NE	12.5	29.2	4.0	7.6	183	NE	NE	NE	NE	NE	NE	NE	NE
	100.00	2,750	1,830	1,750	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
GG-211D.....	.10	NE	NE	NE	9.2	44.6	5.7	8.9	201	457	.55	NE	205	NE	91.5	4.4	NE
	100.00	900	850	640	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
GG-416.....	.05	NE	NE	NE	7.9	8.2	4.9	5.7	301	NE	NE	NE	NE	NE	NE	NE	NE
	.10	NE	NE	NE	9.5	54.6	4.4	12.1	233	536	.54	NE	203	NE	87.4	4.5	NE
	100.00	1,850	1,800	1,730	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
GG-610.....	.05	NE	NE	NE	8.5	5.4	3.5	3.3	242	NE	NE	NE	NE	NE	NE	NE	NE
	.10	NE	NE	NE	9.0	11.3	5.2	8.8	180	564	NE	NE	NE	NE	NE	NE	NE
	100.00	3,210	2,630	2,310	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
GG-813.....	.10	NE	NE	NE	10.5	10.2	5.0	7.4	172	NE	NE	NE	NE	NE	NE	NE	NE
	100.00	1,720	NE	1,180	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
Clay:																	
IV-15C.....	1.00	NE	NE	NE	7.4	2.6	2.0	5.9	496	878	.39	.33	285	314	95.9	2.2	23.5
	100.00	87	NE	94	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
ITS-46.....	1.00	NE	NE	NE	6.6	4.0	3.0	6.8	382	NE	.48	NE	243	NE	93.5	3.9	NE
	100.00	70	65	59	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
WB-17.....	1.00	NE	NE	NE	7.5	9.2	4.1	20.0	527	737	.43	.34	252	288	93.1	2.6	21.9
	100.00	940	850	473	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE	NE
	.75	NE	NE	NE	7.4	3.4	2.6	17.7	404	NE	NE	NE	NE	NE	NE	NE	NE
	.50	NE	NE	NE	7.0	4.2	3.5	10.9	375	927	.56	NE	206	NE	90.8	3.4	NE

See explanatory notes at end of table.

TABLE 11. - PWAT values and pellet properties obtained with different clays and organic mixtures--Continued

Binders	pct	PWAT No.[1] H_2O			Unfired				Fired strength, lb		R_{40},[2] pct/min		t_{90},[3] min		RDI,[4] pct		Porosity, pct
		Pure	Tap	Wash	H_2O, pct	Drop No.	WCS, lb	DCS, lb	[5]1,200	[5]1,250	[5]1,200	[5]1,250	[5]1,200	[5]1,250	Plus 6.3 mm	Minus 0.5 mm	
Organic and clay:																	
PAC.....	0.08	NE	NE	NE	8.1	11.0	3.7	18.4	379	NE	NE	NE	NE	NE	NE	NE	NE
WB-17.....	.75																
PS-200.....	.10	246	NE	252	9.7	4.8	3.9	16.8	581	NE	0.56	NE	256	NE	94.9	2.5	23.3
IV-15-C.....	.50																
GG-211D.....	.10	233	NE	259	7.0	4.8	3.1	8.2	429	NE	.59	NE	198	NE	94.9	3.6	NE
IV-15-C.....	.50																
GG-416.....	.05	271	NE	253	9.0	14.2	5.9	8.4	388	NE	.53	NE	225	NE	NE	NE	NE
IV-15-C.....	.50																
GG-416.....	.10	320	NE	302	10.2	50.4	4.1	11.2	308	654	.56	NE	203	NE	94.8	3.9	26.3
IV-15-C.....	.50																
GG-416.....	.10	NE	NE	NE	7.3	4.6	3.1	10.1	319	605	NE	NE	NE	NE	NE	NE	NE
IV-15-C.....	.50																
GG-610.....	.10	440	NE	463	11.0	28.0	6.1	10.9	326	664	.69	NE	190	NE	92.1	4.8	28.9
IV-15-C.....	.50																
GG-610.....	.10	NE	NE	NE	6.9	3.8	2.9	8.4	470	NE	NE	NE	NE	NE	90.5	3.5	NE
IV-15-C.....	.50																
PS-200.....	.10	190	NE	214	7.5	5.9	2.5	8.5	399	NE	.56	NE	212	NE	NE	NE	25.6
ITS-46.....	.50																
PS-200.....	.30	268	NE	374	7.1	4.3	2.6	22.5	272	707	.74	NE	158	NE	91.6	5.1	NE
ITS-46.....	.50																
PS-300.....	.10	650	NE	319	7.2	3.3	2.5	5.9	386	NE	.53	NE	209	NE	92.1	4.1	25.4
ITS-46.....	.50																
PS-300.....	.30	1,213	NE	493	7.0	4.6	2.4	19.4	288	794	.74	NE	158	NE	90.5	4.0	NE
ITS-46.....	.50																
GG-211D.....	.10	242	NE	272	6.8	3.3	3.2	7.1	354	800	.62	NE	195	NE	92.4	3.6	25.6
ITS-46.....	.50																
GG-416.....	.10	321	NE	321	7.7	3.9	3.0	7.1	304	579	.52	NE	208	NE	NE	NE	NE
ITS-46.....	.50																
GG-610.....	.10	729	NE	397	7.3	4.6	2.8	9.2	381	NE	NE	NE	NE	NE	NE	NE	NE
ITS-46.....	.50																
GG-416.....	.10	270	NE	281	7.5	5.3	3.4	7.4	452	NE	NE	NE	NE	NE	NE	NE	NE
ITS-46.....	.90																

CMC Carboxyl methyl cellulose. ITS Itasca County. PAC Polycrylate. SGC Starch graft copolymer.
DCS Dry compressive strength. IV Area 4 clay. PS Pregelatinized starch. WB Western bentonite.
GG Guar gum. NE Not evaluated. PWAT Plate water absorption test. WCS Wet compressive strength.

[1]Determined with no iron oxide concentrate present; therefore, percentages of samples and organic designate only their relative ratio. Pure, distilled water; tap, city water; wash, filtrate from a 50-pct-solids slurry made with iron oxide concentrate.

[2]Reduction rate at 40 pct reduction.

[3]Time required to obtain 90 pct reduction.

[4]Percent plus 6.3- and minus 0.5-mm particles produced in reduction disintegration index (RDI) tests.

[5] Degree Celsius.

Unfired Pellet Properties

Clay Binders

Commercial Samples

The commercial samples were investigated at the 1-pct addition level to obtain baseline unfired pellet data for comparison purposes. In general, the results in table 12 indicate that the western bentonites had higher dry compressive strengths than did the other materials; the strengths were expressed as the load required to break the pellet. The lowest values of each of the unfired pellet properties of the western bentonites were used to define the minimum acceptable values that would have to be met by any potential binder. The pellet target values were defined as follows:

Dry compressive strength = 14 lb.

Wet compressive strength = 3 lb.

Drop number = 4.

With a few of the western bentonites, the dry strengths were almost twice as high as the target values. The average unfired pellet properties of the western bentonites were fairly well represented by WB-17; this sample was referred to as the typical western bentonite. With the southern bentonite and bentonite-mica samples, these target values were almost reached. The unfired pellet target values were not reached with any of the nonbentonite samples. The nonbentonite materials were selected for certain known properties and/or for being possible constituents in midwestern clays.

Drying Temperature

The typical western bentonite (WB-17) was usually dried overnight at 75° C, but higher temperatures were also used. At the 1-pct binder addition level, more moles of aluminum and silicon oxide were added with the binders when the samples were dried at the higher temperatures, owing to moisture loss (figure 2 and table 5). However, with the higher temperature (600° and 800° C) dried binder, the property values of unfired pellets were lower (table 12). The decreased strength values were possibly due to the collapse of the hydroxylated montmorillonite structure.

TABLE 12. - Properties of unfired pellets made with 1 pct addition of commercial samples

Sample	H_2O, pct	Drop No.	WCS, lb	DCS, lb	Sample	H_2O, pct	Drop No.	WCS, lb	DCS, lb
Chlorite, CH.....	7.2	2.7	2.1	3.4	Western bentonite--Con.				
Meta bentonite, M-B..	7.5	2.7	1.9	3.8	WB-5.....	8.7	10.0	3.2	17.0
Silica gel, SG.....	7.6	3.4	2.6	3.9	WB-6.....	8.1	6.0	2.8	18.0
Zeolite, Z.....	7.5	3.8	2.3	4.1	WB-7.....	7.0	4.5	4.3	18.3
Kaolinite, KA-1.....	7.2	3.9	2.8	4.4	WB-8.....	7.8	6.3	3.6	18.5
Vermiculite, VE.....	7.1	2.7	2.3	5.1	WB-9.....	7.4	5.0	3.5	18.5
Fire clay, KA-2.....	7.2	2.9	2.9	5.3	WB-10.....	7.0	5.9	4.3	19.0
Attapulguite, AT.....	7.1	2.9	2.4	6.0	WB-11.....	8.7	8.5	3.3	19.3
Illite, I-S.....	6.9	3.0	3.4	6.0	WB-12.....	7.0	8.2	5.2	19.9
Al_2O_3-colloidal, Al-C	7.3	3.6	2.6	6.2	WB-13.....	7.4	4.6	3.8	21.2
Beidellite, BE.....	7.1	3.6	2.9	9.4	WB-14.....	8.5	6.3	4.2	21.6
Southern bentonite, SB.....	8.1	5.3	3.0	11.7	WB-15.....	8.2	9.9	3.6	22.7
Bentonite-mica, B-M..	8.6	8.7	2.6	13.8	WB-16.....	7.2	7.6	4.1	24.3
Western bentonite:					WB-17.....	7.6	9.2	4.1	20.0
WB-3.....	7.6	4.0	3.5	14.3	WB-17, 600[1].....	9.6	7.9	3.3	18.7
WB-4.....	7.2	4.7	3.8	16.5	WB-17, 800[1].....	7.2	3.8	3.1	7.0

DCS Dry compressive strength. WCS Wet compressive strength.
[1]Drying temperature (°C); all other samples dried at 75° C.

Midwestern Samples

The initial pelletizing experiments were conducted with split-tube and shovel clay samples from various counties at the 1-pct addition level. The unfired pellet results in figure 5 indicated that the largest dry compressive strengths of the pellets were obtained with the Red River Valley samples from northwestern Minnesota in Clay, Kittson, Marshall, Norman, and Polk Counties, and from northeastern North Dakota in Walsh County. The wet compressive strength and drop numbers showed no particular pattern and were generally below the minimum target values obtained with the western bentonites.

To determine the binder percentage required to reach the minimum western bentonite target level, more extensive tests were conducted with the augered samples. The results in figure 6 and table 13 indicate that the target unfired strength values were reached with 3-pct additions of samples IV-15C and VI-37A. However, this is an extremely high addition level, so attempts were made to improve the binding properties by the addition of soda ash.

Soda Ash Addition

The properties of the unfired pellets made with the auger samples at the 1-pct binder level generally improved with the addition of 0.1 pct soda ash but the target level was still not obtained (fig. 7). The addition of soda ash to southern bentonite considerably improved the unfired pellet properties (fig. 6). These results indicated that soda ash addition improves the unfired properties of samples that contain calcium montmorillonite, probably because the montmorillonite is converted to the more effective sodium form.

Higher binder levels with Minnesota clays and soda ash were investigated to determine conditions required to reach the target unfired strengths. Using 2 pct binder (with area IV-15C clay and soda ash), the unfired pellet properties increased with increasing soda ash additions up to a maximum value. This maximum occurred with 1.94 pct clay and 0.06 pct soda ash; at this addition level, all the unfired pellet target values were reached.

Different soda ash addition methods were investigated to further improve the unfired pellet properties. The first method involved mixing the iron ore concentrate first with the clay and later with the soda ash. The second method involved changing the order of additions of the clay and soda ash and/or the water. The time before pelletizing the wet mixture was also varied from 1/2 h to 3 days. Essentially the same unfired pellet strengths were obtained with all these soda ash treatment methods.

Binder Strength Versus Clay Characteristics

To predict the binder effectiveness, the pellet strength data were compared to the clay characterization data. The results in figure 8 suggested that there was a definite correlation between dry pellet strength and some of the clay characterization variables. Correlation coefficients were calculated assuming a linear relationship between these variables. The highest correlation coefficients were assigned the lowest ranking numbers (table 14). These results indicated that the PWAT number and CEC-mb clay characterization variables were the best indicators of the dry pellet strengths.

Not all pellets with high dry strengths had high drop numbers (fig. 9). The clay characterization variable that resulted in the highest correlation coefficient for the drop numbers was the viscosity of a 6-pct binder slurry (table 14).

Organic and/or Clay Binder Mixtures

The unfired pellet properties generally were improved by the use of organic binders, which is in agreement with results reported by other investigators (16-18). With 0.3 pct organic binder, such as pregelatinized starches (PS-200 and PS-300), high dry compressive strengths of 41.8 and 19.6 lb, respectively, were obtained (table 11). When the organic additions were decreased to the 0.1-pct

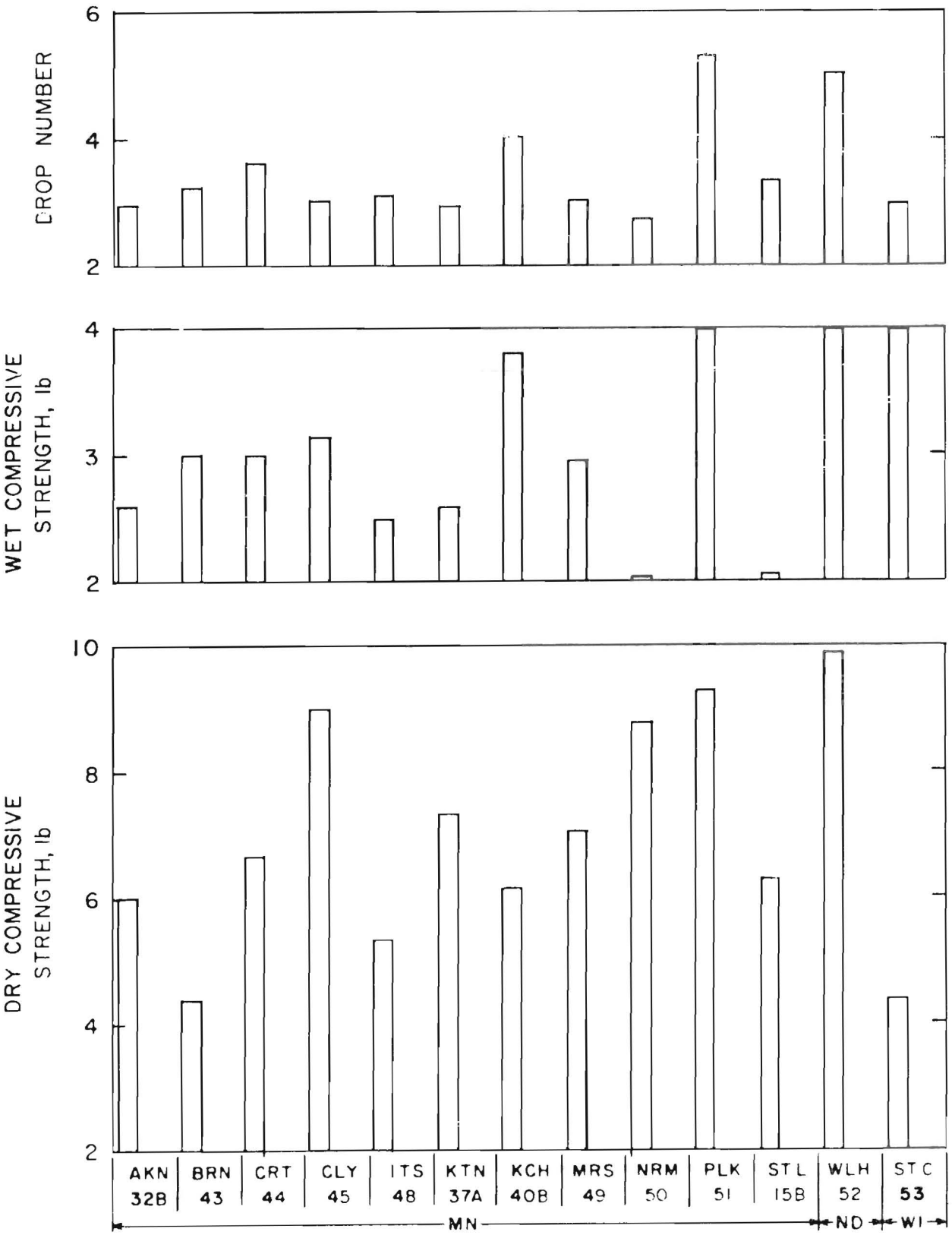

FIGURE 5.—Properties of unfired pellets made with 1 pct additions of auger, split-tube, and shovel clay binders from various counties (table 1) in Minnesota, North Dakota, and Wisconsin.

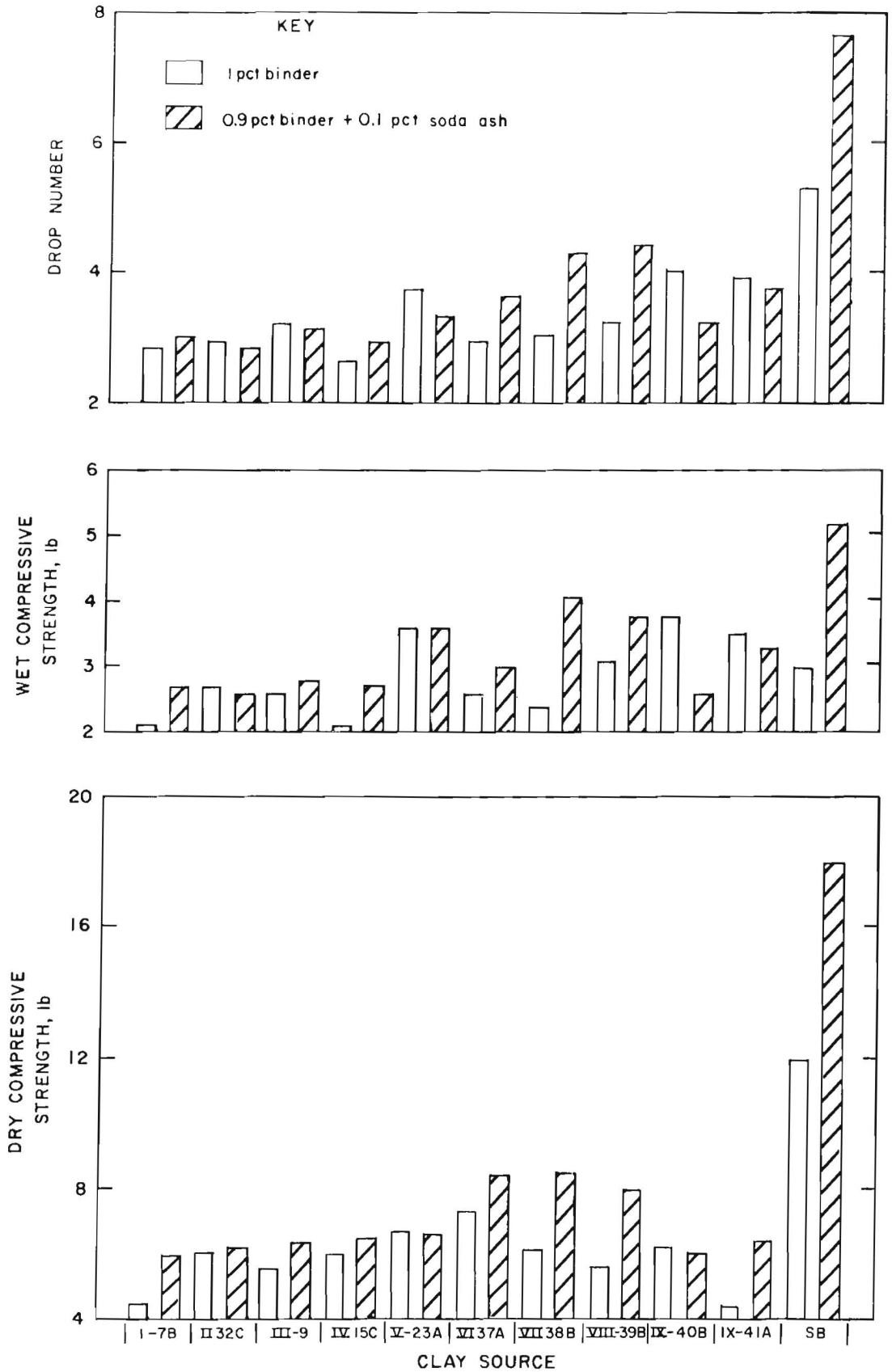

FIGURE 6.—Influence of clay binder concentration on properties of unfired pellets.

TABLE 13. - Physical and metallurgical properties of pellets made from selected augered Minnesota clay samples, with and without soda ash

(Reduction at 900° C with 30 pct CO and 0.2 pct H_2 in N_2 at 1.5 SLM)

Sample and pct	Soda ash, pct	Unfired H_2O, pct	Unfired Drop No.	Unfired WCS, lb	Unfired DCS, lb	Fired strength, lb [3]1,200	Fired strength, lb [3]1,250	R_{40},[1] pct/min [3]1,200	R_{40},[1] pct/min [3]1,250	t_{90},[2] min [3]1,200	t_{90},[2] min [3]1,250
I-7B:											
1.00	0.00	7.4	3.0	2.2	4.4	460	698	NE	0.38	NE	282
3.00		7.5	2.8	2.2	7.1	458	634	NE	NE	NE	NE
0.99	.01	6.7	3.0	3.0	5.8	342	630	0.49	NE	260	NE
0.90	.10	6.7	3.0	3.0	5.8	430	663	NE	NE	NE	NE
0.90	.10	7.3	3.0	2.7	5.9	381	NE	NE	NE	NE	NE
II-32C:											
1.00	0	7.3	2.9	2.7	6.0	387	727	NE	.35	NE	293
2.00	0	6.7	5.7	4.0	9.6	509	NE	.34	NE	333	NE
0.90	.10	7.2	2.8	2.6	6.1	413	623	.49	NE	260	NE
III-98:											
1.00	0	7.3	3.2	2.6	5.5	539	667	NE	.29	NE	358
0.90	.10	7.2	3.1	2.8	6.3	299	871	NE	NE	NE	NE
IV-15C:											
0.10	0	7.1	2.9	2.5	5.5	381	NE	NE	NE	NE	NE
1.00	0	7.4	2.6	2.0	6.0	422	739	.39	.33	285	314
2.00	0	7.0	2.8	2.2	9.0	465	637	.51	NE	261	NE
3.00	0	7.4	5.7	4.0	14.8	574	664	NE	NE	NE	NE
5.00	0	7.0	10.1	4.1	22.8	633	NE	NE	NE	NE	NE
0.90	.10	7.4	2.9	2.7	6.4	336	584	NE	NE	NE	NE
1.98	.02	7.0	3.1	2.8	8.8	429	651	NE	NE	NE	NE
1.94	.06	6.9	6.2	4.4	14.3	731	NE	.45	NE	307	NE
1.86	.14	7.2	6.6	4.1	13.1	656	NE	NE	NE	NE	NE
1.80	.20	7.4	5.0	3.0	11.4	429	NE	.46	NE	291	NE
V-23A:											
1.00	0	7.1	3.7	3.6	6.7	523	658	NE	.32	NE	302
0.90	.10	6.6	3.3	3.6	6.6	465	751	NE	NE	NE	NE
VI-37A:											
1.00	0	7.2	2.9	2.6	7.3	477	562	.54	.47	232	258
2.00	0	7.6	4.3	2.1	11.4	546	NE	.54	NE	248	NE
3.00	0	6.8	7.4	3.8	17.1	730	NE	NE	NE	NE	NE
0.90	.10	7.2	3.6	3.0	8.7	460	708	.53	NE	267	NE
VII-38A:											
1.00	0	7.2	3.0	2.4	6.1	422	NE	NE	NE	NE	NE
0.90	.10	6.5	4.3	4.0	8.5	608	NE	NE	NE	NE	NE
VII-39A:											
1.00	0	7.0	3.2	3.1	5.6	643	NE	NE	NE	NE	NE
0.90	.10	6.8	4.4	3.8	8.0	601	NE	NE	NE	NE	NE
IX-40A:											
1.00	0	7.0	4.0	3.8	6.2	606	NE	NE	NE	NE	NE
0.90	.10	7.4	3.2	2.6	6.0	475	NE	NE	NE	NE	NE
IX-41A:											
1.00	0	7.4	3.9	3.5	4.3	504	NE	NE	NE	NE	NE
0.90	.10	7.4	3.8	3.3	6.4	525	NE	NE	NE	NE	NE
WB-17:[4] 1.00	0	7.6	9.2	4.1	20.0	527	737	.43	.34	252	288
WB-600:[5] 1.00	0	8.2	7.9	3.3	18.7	557	NE	NE	NE	NE	NE
WB-800:[6] 1.00	0	7.2	3.8	3.1	7.0	326	NE	NE	NE	NE	NE
Na_2CO_3: 0	.20	4.3	3.7	4.0	7.7	527	NE	NE	NE	NE	NE
No additives: 0	0	6.3	3.2	3.1	2.3	284	375	.80	.69	150	178

DSC Dry compressive strength.
NE Not evaluated.
WCS Wet compressive strength.
[1]Rate at 40 pct reduction.
[2]Time required to obtain 90 pct reduction.
[3]Degree Celsius.
[4]Dried at 75° C.
[5]Dried at 600° C.
[6]Dried at 800° C.

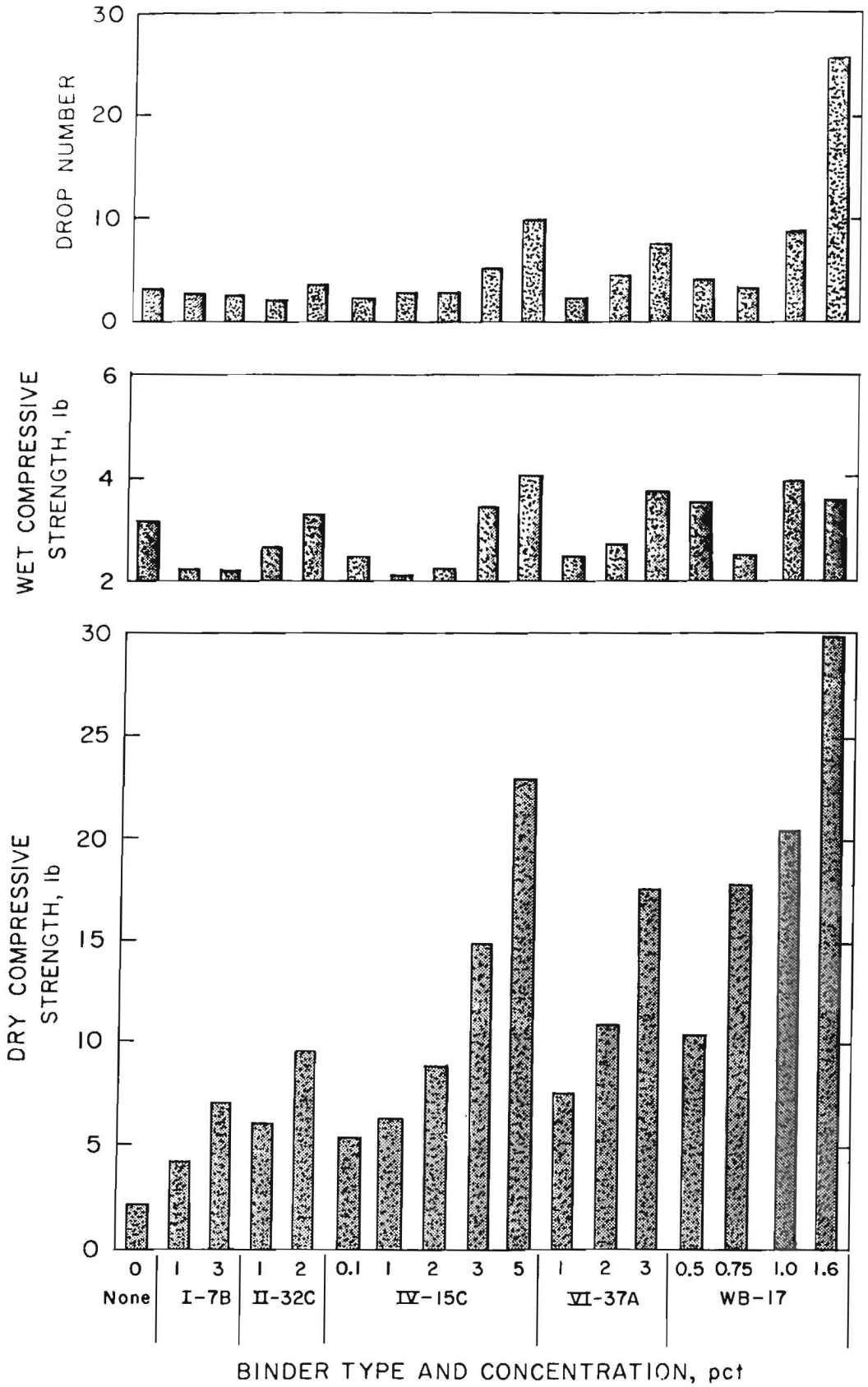

FIGURE 7.—Effect of the clay binder source, with and without soda ash, on properties of unfired pellets.

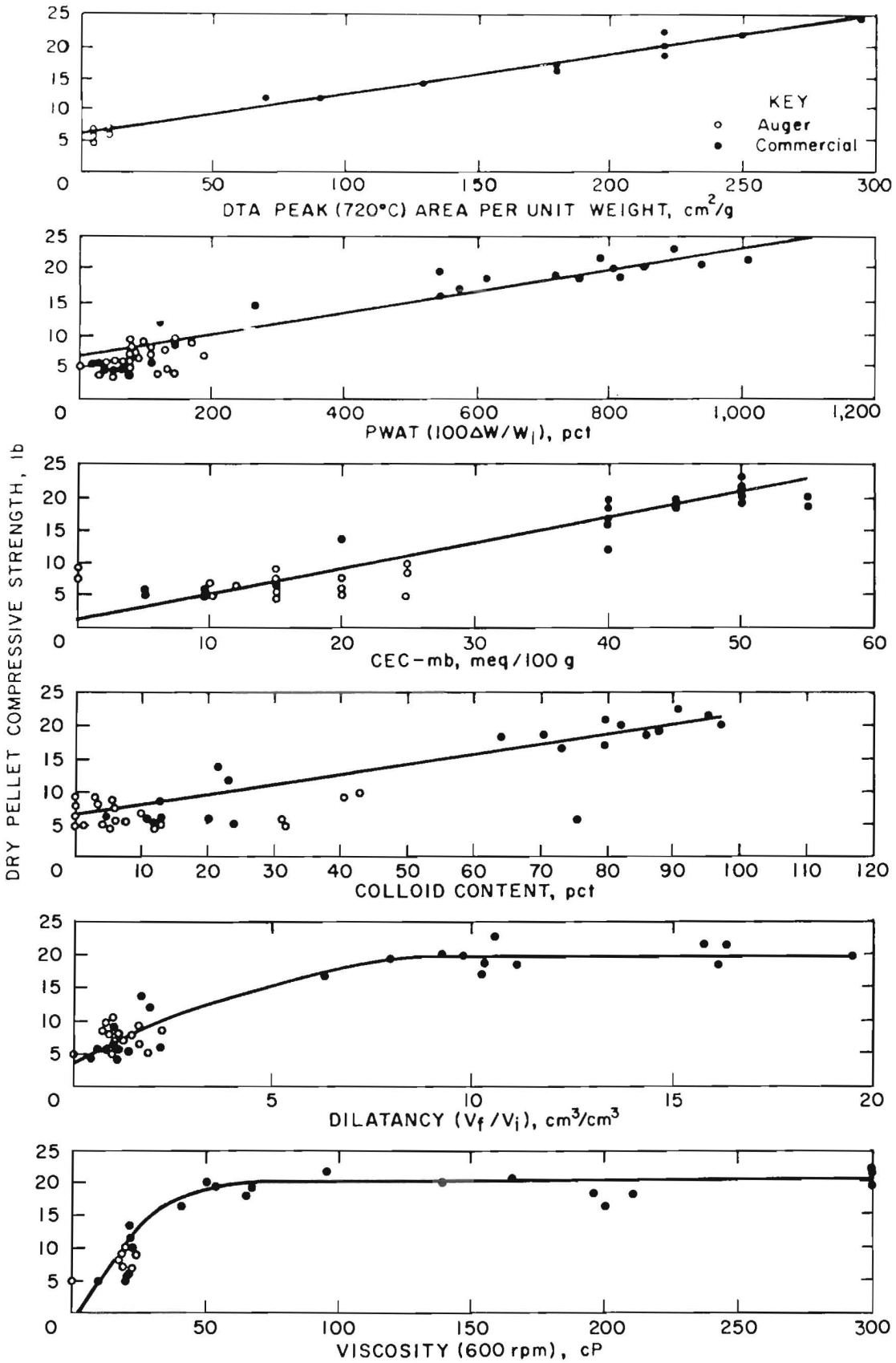

FIGURE 8.—Dependence of clay characterization variables on the dry, unfired pellet strength.

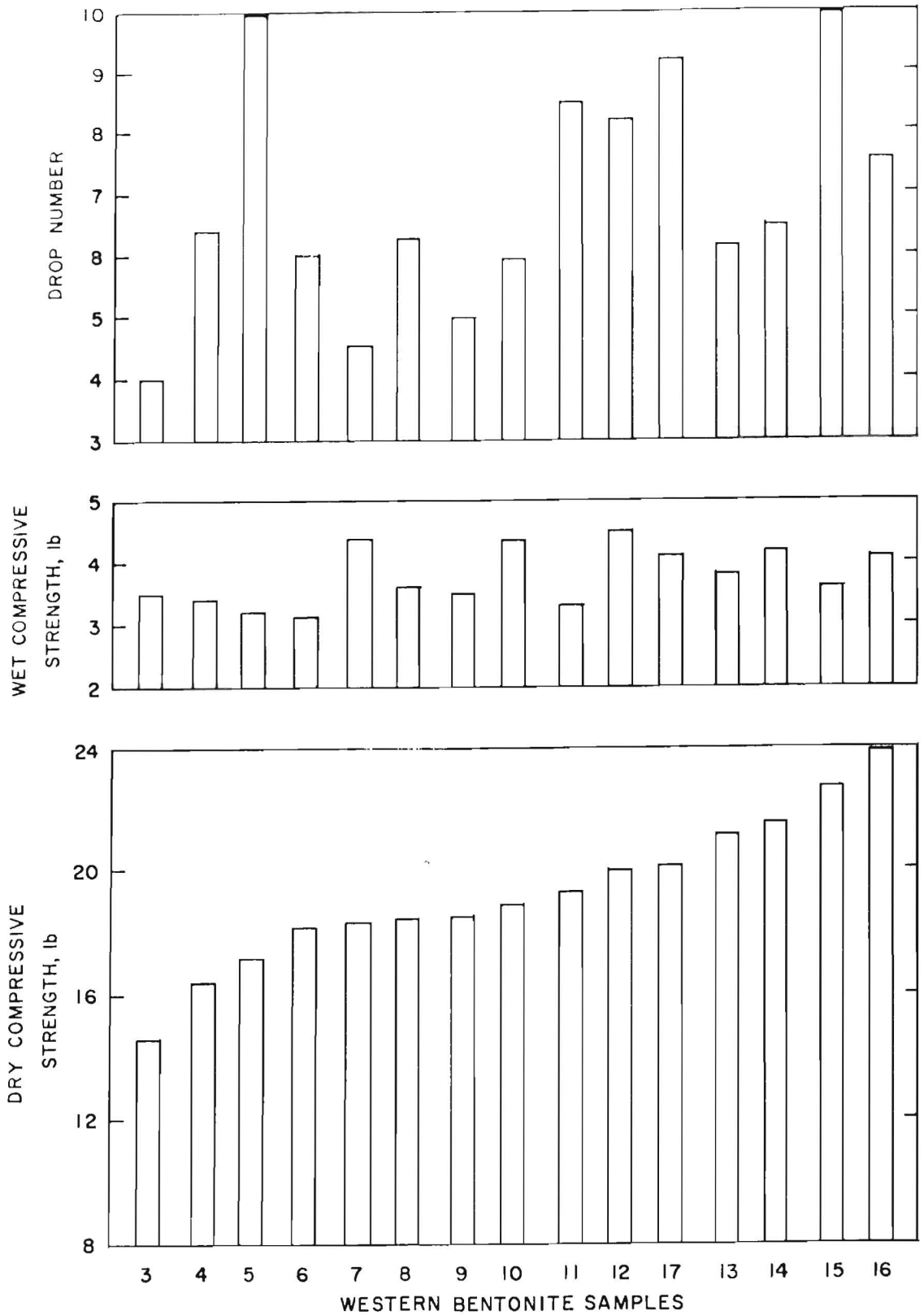

FIGURE 9.—Influence of different western bentonites, at 1 pct addition level, on properties of unfired pellets.

level, smaller improvements in the pellet properties were observed. For example, with PS-200, the dry compressive strength was only 11.3 lb, which was less than the target value. Starch-graft copolymers (SGC) had higher PWAT values than the pregelatinized starch binders but the pellets had low dry compressive strengths. Therefore, PWAT values were not the best indicator of relative binder effectiveness of organic materials.

When the organic materials were added to Minnesota clays, both the PWAT numbers and dry pellet strengths increased (table 11). For example, with a 0.5 pct IV-15C or ITS-46 and 0.1 pct PS-200 binder mixture, a dry compressive strength comparable to that with 0.5 pct western bentonite (WB-17) was obtained. The paint rock sample (ITS-46) was preferred because it contained only 3.4 pct Si; the other clay samples contained over 20 pct Si. The silica content of the binder is especially important in a direct-reduced iron process (19).

In general, the pellet drop numbers also were higher when the organic materials were added to clay. For example, when 0.08 pct polyacrylate (PAC) was added to 0.75 pct western bentonite binder, the drop numbers increased from 3.4 to 11.0 (table 11). The viscosities increased considerably when polyacrylate (PAC) or guar gum (GG) were added to the typical western bentonite (table 10). These results are consistent with the clay characteristics correlation data, which indicated that the drop numbers were most dependent on the binder slurry viscosity (table 14).

The drop numbers were also dependent on the water content of the pellets. The results (table 11) showed that the drop number increased considerably at the higher moisture levels, as indicated by the mixture of 0.5 pct IV-15C and 0.1 pct GG-416. Because dry concentrate was used in this laboratory research, less than 8 pct water was usually present in the wet pellets; this moisture level was slightly lower than that used in commercial pelletizing plants, which usually operate at pellet moisture levels of about 9 pct. With the laboratory method, more pelletizing problems were encountered at the higher moisture levels.

Fired Pellet Properties

Clay Binders

The fired pellet properties were evaluated by the fired strengths, porosity, thermal shock test, reducibility, and reduction disintegration index as described in appendix C. The greatest emphasis was placed on producing pellets with high reducibility and target fired strengths

TABLE 14. - Ranking of clay characterization variables and pellet properties via correlation coefficients (cc)[1]

Variable	Drop No.		WCS, lb		DCS, lb		Variable	Drop No.		WCS, lb		DCS, lb	
	cc	Rank	cc	Rank	cc	Rank		cc	Rank	cc	Rank	cc	Rank
CEC:							DTA peak						
mb.....	0.64	2	0.62	8	0.95	1	(720° C).	0.63	4	0.83	1	0.89	4
Na^+ BTAC	.51	9	.77	2	.86	6	Dilatancy.	.59	6	.72	4	.87	5
Electrode:							PWAT:						
Na^+.....	.54	8	.75	3	.77	9	Swell...	.57	7	.68	5	.91	3
Ca^+.....	.50	10	.48	10	.71	10	No.....	.63	3	.68	6	.94	2
Eh.....	.31	11	.40	12	.55	11	LOI (260°-						
pH.....	.27	12	.41	11	.54	12	105° C)..	.03	15	.15	14	.04	15
Colloids:							Si-Al ratio	.06	14	.09	15	.15	14
Fine....	.26	13	.32	13	.40	13	Viscosity						
Total...	.62	5	.67	7	.82	7	(600 rpm).	.73	1	.58	9	.78	8

DCS Dry compressive strength. WCS Wet compressive strength.

[1]Lowest rank number assigned to variable with highest cc. Coefficients were determined assuming a linear relationship existed between clay characterization variable and pellet properties.

of over 330 lb. The shock test was conducted at the same conditions (1/2 h at 900° C) that were used in the first stage of the pellet induration procedure as described in appendix C. The only pellets that broke apart in this test were those made with the typical western bentonite (WB-17) dried at 600° C or higher temperatures; more than 60 pct of the pellets broke apart in the shock test with pellets made with 1 pct of this bentonite dried at these high temperatures.

The reduction tests were conducted at 900° C with 30 pct CO and 0.2 pct H_2 according to the most recent ISO standard (20). The total gas flow rate was 1.5 SLM. A single pellet was used in these tests as described in reference 21. The relative reduction was determined by both the time required to reach 90 pct reduction (calculated from the weight loss data) and the reduction rate (dR/dt) at the 40-pct reduction level as determined by

$$\left[\frac{dR}{dt}\right]_{40} = R_{40} = \frac{33.6}{(t_{60} - t_{30})}, \qquad (1)$$

where t_{60} and t_{30} refer to the times, in minutes, required to obtain 60- and 30-pct reduction, respectively. Some tests were conducted with the older ISO method (22) using 950° C and 40 pct CO. This resulted in R_{40} values that were about twice as large as those obtained at 900° C and 30 pct CO. The strong dependence of the reduction rate on temperature and reductant concentration was consistent with previous research conducted in this laboratory (21).

The results in figure 10 indicate that the addition of 1 pct Minnesota clay to iron oxide concentrate lowered the reduction rate of the pellets considerably. When no binder was added to the pellet, the R_{40} value was high (table 13), but the R_{40} values decreased as the quantity of clay was increased. The highest reduction rates were obtained with an area VI sample and the lowest with an area III sample. The typical western bentonite (WB-17) had an R_{40} value between samples VI-37A and IV-15C (table 13). The precise reasons for the lower reduction

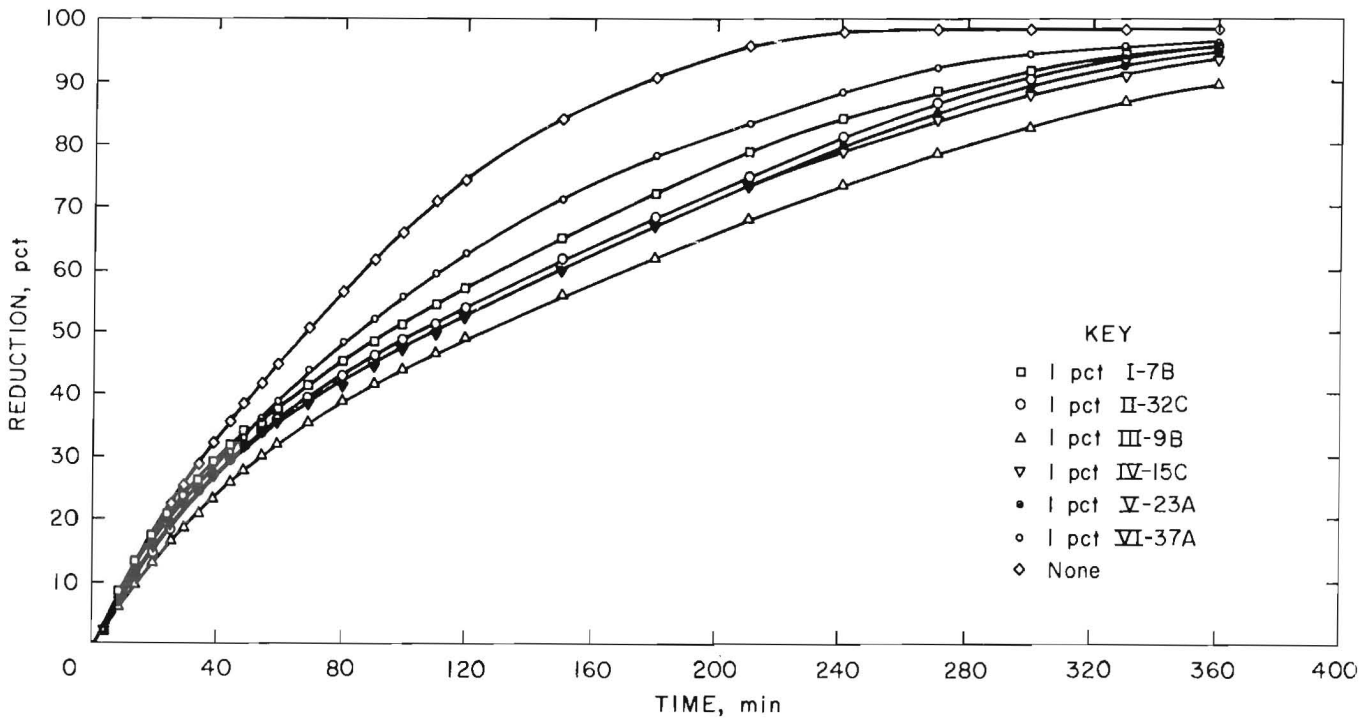

FIGURE 10.—Influence of different Minnesota clay binders on reduction of pellets. (Pellets were fired at 1,250° C and reduced at 900° C with 30 pct CO and 0.2 pct H_2 in N_2 a 1.5-SLM flow rate.)

rates when different samples were added were not known, but one of the reasons was probably the presence of low-melting constituents in the samples. This observation was also supported by the decrease in R_{40} when a low-melting compound, soda ash, was added.

For pellets indurated for 15 min in a muffle furnace at 1,200° C, the target fired compressive strength of 330 lb was obtained with all the Minnesota clay samples. With no binder, the target strength was reached only when the temperature was increased to 1,250° C; however, this also decreased the R_{40} value.

Organic Binders

In general, when organic materials were used as a binder, the fired strengths were much lower than with the Minnesota clay or western bentonite samples and target strengths were not obtained at 1,200° C (table 11). The fired strengths decreased with increasing amounts of organic additives. Target strengths were obtained by increasing the firing temperature to 1,250° C, but unfortunately, the reduction rates also decreased.

Organic and Clay Binder Mixtures

The binder mixture studies were conducted with the organic materials that contained less than 3 pct sodium. The low-sodium binders were preferred because sodium is known to be harmful to blast furnace refractories.

When 0.1 pct organic materials (except guar gum 416) were added to 0.5 pct Minnesota clay samples, fired (1,200° C) strengths of over 330 lb were obtained (table 11). Also, the reduction rates were higher than with Minnesota clay binder alone, which is consistent with the generally higher porosities obtained with the mixtures.

The percentage of the plus 6.35-mm tumble fraction from the reduction disintegration index test generally was also higher with the mixtures of 0.1 pct organic material and 0.5 pct Minnesota clay samples than with straight organic binders. In addition, the quantity of fines (minus 0.5 mm) was generally lower with the mixtures than with the straight organic materials.

SUMMARY AND CONCLUSIONS

An extensive clay-sampling and characterization investigation was conducted on clays from former glacial lakes in the Midwest, mainly those in Minnesota. Four glacial lakes were sampled in nine different areas. The sampling consisted of augering 15-cm-diam holes up to depths of 22 m. A total footage of 450 m was obtained from 40 holes. One-hundred-fifty 1.5-m sample sections were selected for the laboratory studies. Most of the footage contained clay- and silt-sized particles, but most of the minerals were from igneous and metamorphic rocks abraded by the glacier. Only a small quantity of the clay-sized particles were clay minerals (phyllosilicates), and those included chlorite, kaolinite, and occasionally smectite. Considerably more smectite was found in conventional western bentonite binders, which were studied for comparison purposes. With the Midwest samples, the most smectite occurred in the Red River Valley samples, and these samples had the best binding properties for agglomerating iron ore concentrates.

Fifteen clay characterization tests were conducted on the 30 midwestern clays and 15 western bentonites; some clay characteristics correlated better than others with the pellet binding properties. The cation exchange capacity, using the methylene blue procedure, and the plate water absorption test were the best clay characterization techniques for predicting the efficiency of the clay sample for making pellets with high dry

compressive strengths. This cation exchange capacity method was faster than the plate water absorption test, because several clay samples were evaluated in less than 1 h. The clay slurry viscosity method was the best test for determining the binder that produced the pellets with the highest drop numbers. The clay characterization tests were the most meaningful with clay samples that met the target strengths at the 1-pct addition level.

Adequate fired strengths were obtained with 1-pct Minnesota clay, and reduction rates of the pellets were similar to those made with 1-pct western bentonites. However, about 2 pct of this sample was required to produce similar unfired pellet properties as obtained with 0.5 pct of a typical western bentonite. A small improvement in the binder properties was obtained by the addition of soda ash to Minnesota clay.

The best combined physical and metallurgical pellet properties were obtained when organic materials were mixed with the Minnesota samples. With a mixture of 0.5 pct Minnesota clay (IV-15C) or paint rock (ITS-46) and 0.1 pct pregelatinized starch (PS-200), the unfired pellet properties were about the same as with 0.5 pct of a typical western bentonite. With pellets made using clay and organic mixtures, about the same reduction rates and percentages of plus 6.35-mm particles (reduction disintegration index) were obtained as with a typical western bentonite. The Mesabi paint rock sample (ITS-46) was preferred because it contained only one-fifth of the silica as compared to western bentonite and several times more iron units.

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APPENDIX A.--CLAY CHARACTERIZATION PROCEDURES

CATION EXCHANGE CAPACITY MEASUREMENTS

The cation exchange capacity was determined by two methods. The first method, involving methylene blue dye, has been well documented (14),[1] and only the minor deviations will be decribed here. A 0.2-g dried sample was used rather than 2 cm^3 clay mud. The cation exchange capacity (meq/100 g clay) was calculated by multiplying the milliequivalents of dye absorbed by 100 and dividing by the dry weight of the sample. The other method involved adding 5 g dried sample to 150 cm^3 of a 6-pct solution of benzyltrimethyl ammonium chloride (BTAC). The slurry was mixed for 5 min in a blender at 14,000 rpm and then filtered. The clear filtrate was analyzed for Na^+, K^+, Mg^{2+}, and Ca^{2+}. From these values, the exchange capacity for each ion was calculated.

COLLOID CONTENT DETERMINATION

A 10-g dried sample was mixed with 500 cm^3 distilled water for 5 min in a non-aerating blender at 14,000 rpm. The slurry was then placed in a 500-cm^3 graduate and allowed to settle. After 18 h, the suspended material was removed by suction, and the sediment was dried and weighed. The percent of solids remaining in suspension was referred to as the total colloid content. The suspension was allowed to settle for 6 additional days, and this sediment was referred to as the coarse colloid. This suspension was centrifuged at 5,000 rpm for 15 min, and the percent solids removed were referred to as the fine colloid content.

ELECTRODE MEASUREMENTS

The Eh, pH, Ca^{2+}, and Na^+ values were measured with pH, specific ion, and calomel electrodes. The suspensions from the centrifuged samples, described in the colloid measurement procedure, were used for these tests.

PLATE WATER ABSORPTION TEST

The procedure used in this research for the plate water absorption test was essentially the same as the ASTM method (8). In general, only the procedure modifications will be described here. A sintered alumina plate was placed in the center of a plastic pan, and water was added to the pan until its level was within 6 mm from the top of the plate. Distilled water was used as a standard, but Minneapolis, MN, tap water and the filtrate from an iron oxide concentrate wash solution prepared at 50-pct solids also were used. In the tests involving the clay materials, a 2-g sample (dried at 105° C) was placed on a 9-cm-diam hardened filter paper; this drying temperature was higher than the 75° C that was used in all the other clay characterization tests. The sample was spread out evenly on the filter paper within a 5-cm-diam plastic ring template, and then the template was removed. With some organic materials, only 0.2 g sample was used, because with larger quantities the wet sample expanded beyond the edge of the filter paper. Also with the organic samples, the template was not removed in order to retain the wet material on the filter paper. The sample percentage weight gain (PWAT number) was determined after 18 h on the alumina plate.

SWELLING MEASUREMENTS

In the dilatancy procedure, a 5-g dried sample was added to a small (10-cm^3) graduate; this graduate was tapped lightly five times on a flat surface to settle the sample, and then the dry volume was measured. A large (250-cm^3) graduate was filled with 250 cm^3 distilled water and a small quantity of sample was sprinkled on top of the water, just enough to lightly cover the center portion. The sample was

[1]Underlined numbers in parentheses refer to items in the list of references preceding this appendix.

kept away from the edge of the graduate and allowed to settle to the bottom before more sample was added. This procedure was continued until all of the 5-g sample was added and settled. The wet expanded volume of the sample in the graduate was then noted. The dilatancy value was obtained by dividing the wet expanded gel volume by the dry volume.

The other swelling procedure (PWAT-V) involved measuring the volume of the 18-h gel from the PWAT number test (described in the next section). After that test was completed, the gel was placed in a 25-cm^3 pharmaceutical graduate and its volume measured. The PWAT-V value was calculated by dividing the wet gel volume by the dry volume of an equivalent quantity of sample.

VISCOSITY MEASUREMENTS

The viscosity procedure was similar to the American Petroleum Institute (API) method (15). A slurry containing a 6-pct sample (by weight) was mixed for 5 min at 14,000 rpm in a blender with a nonaeration impeller; the API method specified a mixing time of 20 min, but 5 min was found satisfactory. Immediately after mixing, the slurry was placed in the stainless steel cup provided with the Fann viscometer. The viscometer rotor was then immersed into the slurry, and the cup height was adjusted to the correct level. Viscometer dial readings were taken at 600 rpm, and the viscosity value in centipoise was determined by multiplying the viscometer dial reading by 0.5.

APPENDIX B.--UNFIRED PELLET PREPARATION AND TESTING

RAW MATERIAL PREPARATIONS

All binder samples for the pelletizing tests were dried at 75° C overnight. The auger and split-tube clay samples were pulverized in a Raymond mill containing a screen with 0.2-mm openings. The magnetic iron ore concentrate was dried at 105° C overnight. Then 3 kg of concentrate and a predetermined weight of binder were blended. This composite was first dry blended in a Muller mixer for 30 min and then blended another 30 min while dripping in 390 cm^3 of distilled water. The wet mixture was pushed through a 4-mesh screen. A 60-g portion of the plus 6-mesh fraction was removed for seed pellets. The remaining material was pushed through a 10-mesh screen for use in the pellet growing operation.

PELLET-GROWING PROCEDURE

The seed pellets were placed in the balling drum (40-cm-diam) rotating at about 50 rpm and sprayed with water as needed. Sufficient minus 10-mesh material was added slowly to the growing pellets to always maintain some excess. This fine material was added until pellet diameters were minus 1/2 and plus 7/16 in. Pellets were screened continually to achieve this size. The process of growing pellets was continuous until the minus 10-mesh material was entirely used.

PELLET TESTING

Twenty wet pellets were tested immediately after balling for both the 45-cm drop number and the wet compressive strength. Another 20 wet pellets were placed in an oven at 105° C overnight, and the weight loss was determined; the remaining wet pellets were stored in a closed container. Wet and dry compressive strengths were obtained with a compression machine set at deformation rate of 3.1 cm/min.

APPENDIX C.--FIRED PELLET PREPARATION AND TESTING

PELLET FIRING

From each pelletizing batch, about 40 wet pellets were dried overnight at 105° C. Three batches of pellets were placed on an alumina tray (25 by 12 by 5 cm), and each batch was separated by an alumina spacer. The loaded alumina tray was placed in an electric muffle furnace preheated to 900° C. The inside dimensions of the heated zone was 50 by 20 by 14 cm. The pellets were heated in this furnace for 30 min, and then immediately transferred to a second muffle furnace preheated to a predetermined temperature between 1,150° and 1,350° C; the most commonly used temperature for this furnace was 1,200° C. Both furnaces were continuously flushed with air at 6 SLM. The pellets were left in the second furnace for 15 min and then placed in a sand box for cooling. The loaded tray was covered with a preheated empty tray, which slowed down the pellet cooling rate. The cold pellets were examined to determine the number broken and cracked. The severity of cracking was also recorded. The fired compressive strength of 20 pellets was determined at a 1.9-cm/min deformation rate.

SHOCK TEMPERATURE TEST

This decrepitation procedure consisted of preheating the muffle furnace to the desired temperature (usually 900° C) and inserting an alumina tray containing a single layer of wet pellets. After 30 min at temperature, the tray was removed and the percentage of pellets showing no fragmentation (survivors) was recorded. The furnace was continuously flushed with air at 6 SLM.

LOW-TEMPERATURE BREAKAGE (LTB)

A sample of 0.75 kg wet pellets was dried at 105° C overnight. The dry pellets were then fired in perforated (about two dozen 6-mm holes) alumina crucibles (7-cm ID and 10-cm height) at 900° C for 30 min in an electric muffle furnace and then immediately transferred to a 1,200° C furnace for 15 min. Both furnace muffle chambers were continuously flushed with air at 6 SLM. Fired pellets were sieved on 16.0-, 12.5-, 11.1-, 9.52-, 6.35-, 3.36-, and 0.50-mm screens, and the mass of each portion was recorded. A 0.5-kg sample was composed of plus 7/16-, minus 1/2-in-diam pellets randomly selected for the low-temperature breakage test. The pellets were placed on a perforated (1-mm holes) iron plate in the center of an alumina tube (4-cm ID by 80 cm long). The bottom portion of the tube (preheater) contained alumina beads (8-cm diam) and a few pieces of fire brick. The thermocouple touched the bottom of the iron plate. Both ends of the tube were capped, and the pellet bed was heated by a vertical tube electric furnace. Nitrogen was passed through the tube at 20 SLM until the sample reached 500° C. The nitrogen gas was then turned off and 20-SLM reducing gas mixture was added; the reducing gas contained carbon monoxide, 20±0.5 pct; carbon dioxide, 20±0.5 pct; hydrogen, 2.0±0.5 pct; and nitrogen, balance. After 60 min, the reducing gas was shut off and the tube was cooled to below 100° C with a flow of nitrogen at 3 SLM. The sample was removed from the tube, and the mass of reduced sample was determined. The sample was then placed in a tumbler drum and rotated for a total of 300 revolutions at a rate

of 30±1 rpm. The tumbler drum was 13 cm in ID and 20 cm long and contained two lifters (2 cm wide). The tumbled samples were screened on sieves of 16.0, 12.5, 11.1, 9.52, 6.35, 3.36, and 0.50 mm. The mass of each screen fraction was determined. The reduction disintegration index (RDI), in percent, was calculated from the following formulas:

$$\text{RDI (plus 6.35 mm)} = \frac{m_1 \times 100}{m_0}$$

and

$$\text{RDI (minus 0.5 mm)} = \frac{[m_0 - (m_1 + m_2 + m_3)] \times 100}{m_0},$$

where m_0 = total mass after reduction,

m_1 = mass of plus 6.35-mm material,

m_2 = mass of minus 6.35-, plus 3.36-mm material,

and m_3 = mass of minus 3.36-, plus 0.5-mm material.

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